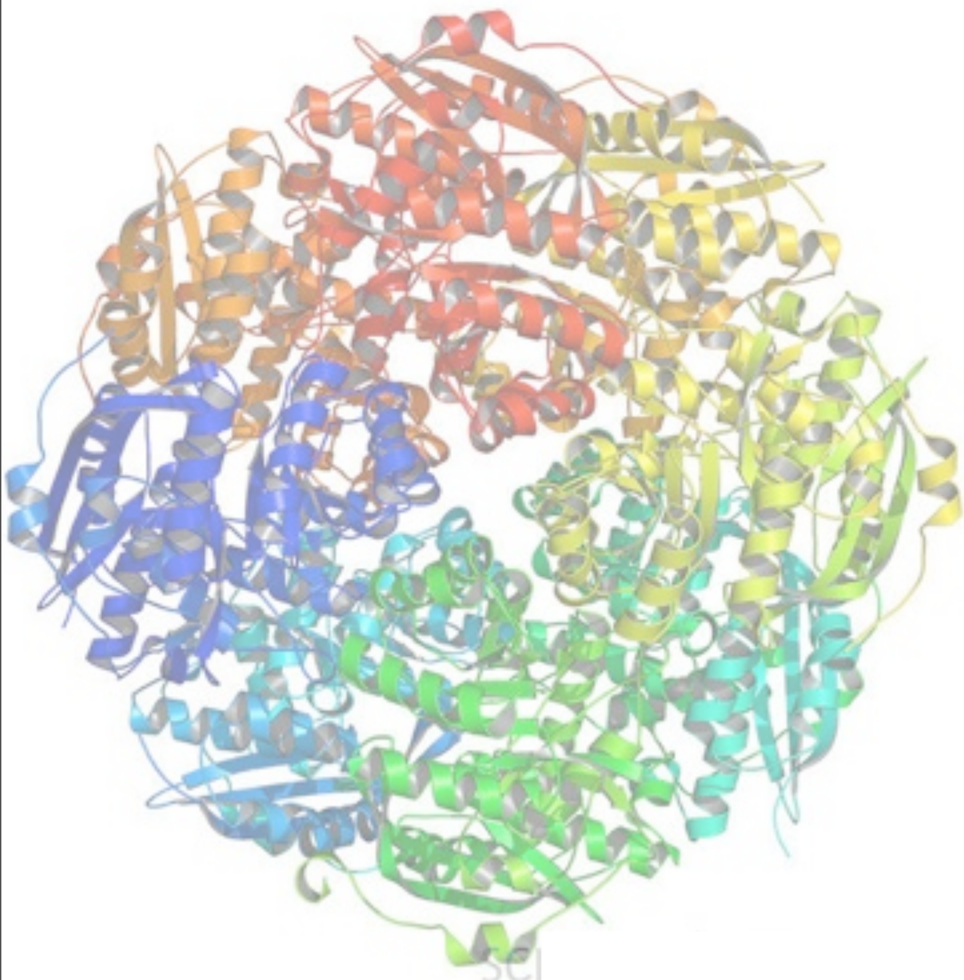

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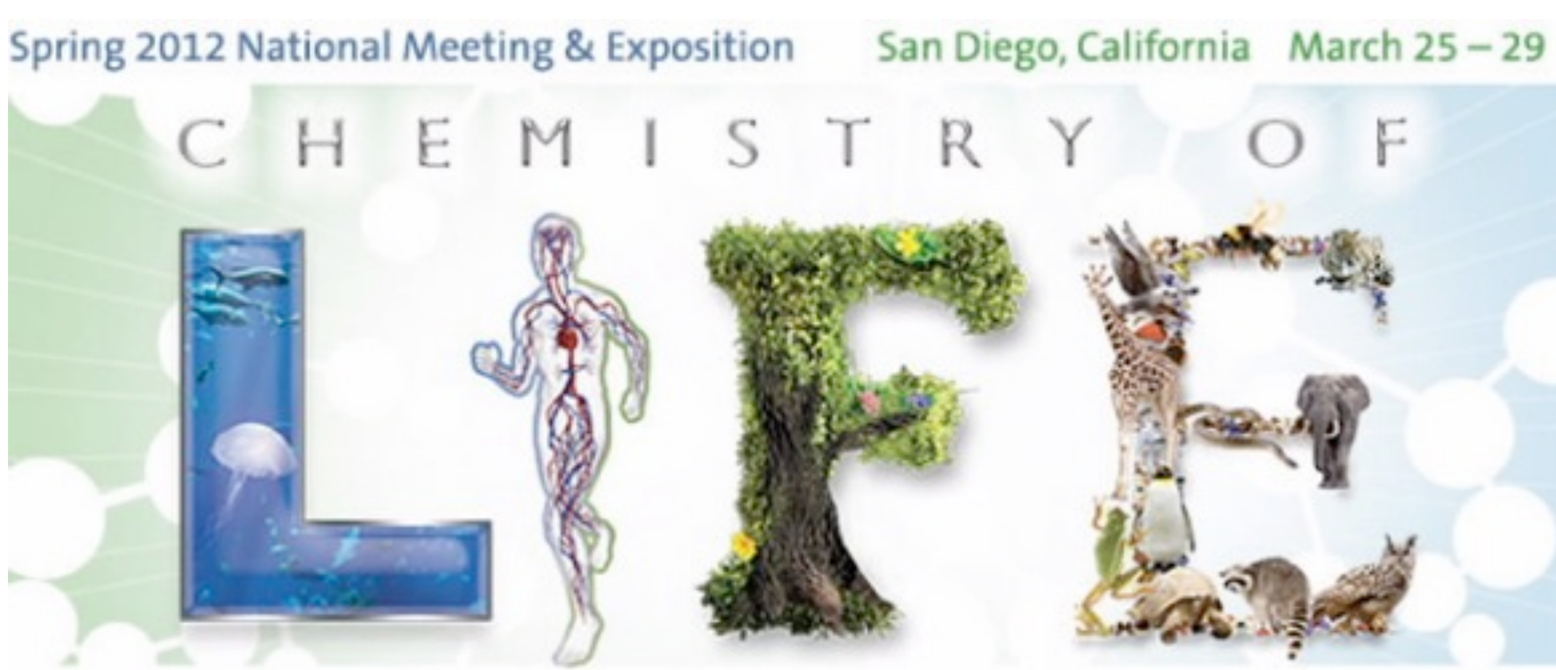
Ionic Liquids For Enzymatic Sensing

29/03/12



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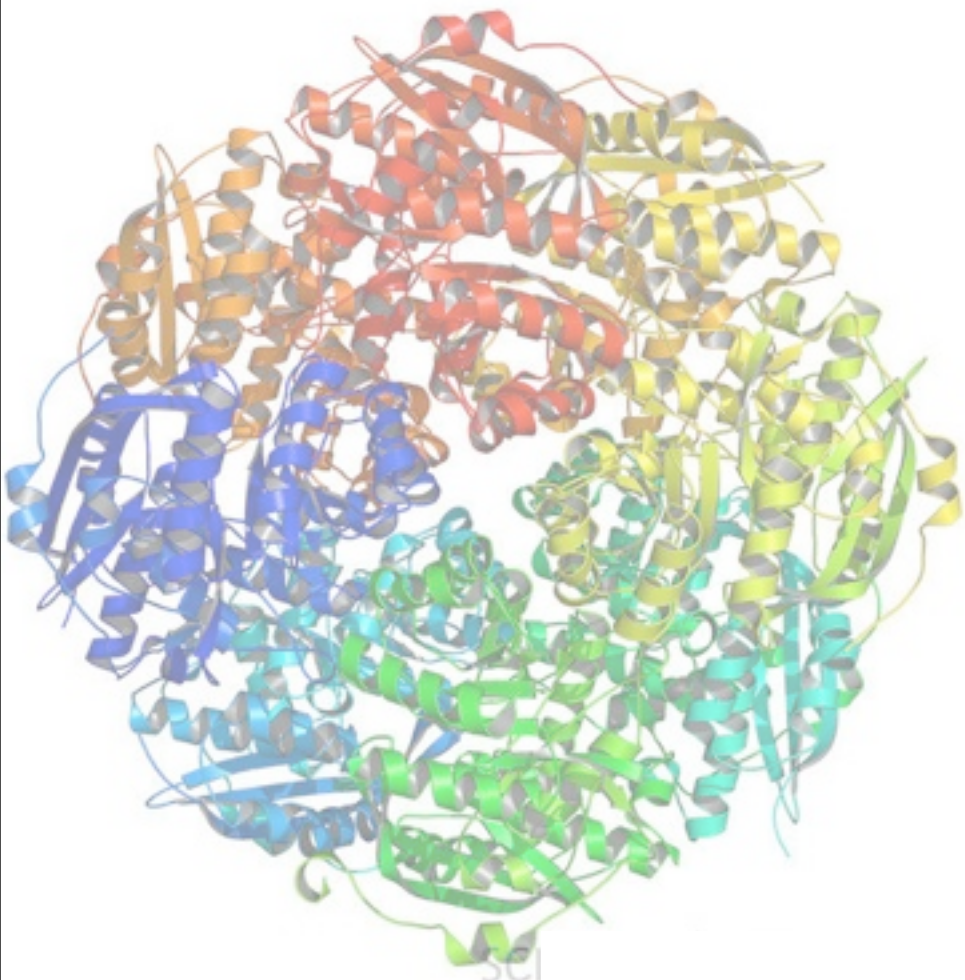

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Ionic Liquids For Enzymatic Sensing

29/03/12

@ cyrusmekon
#ACS

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Contents

- The need for wearable sensors.
- Wearable electrochemical sensors.
- Diverse material for sensing platforms.
  - Ionic Liquids & Organic Electrochemical Transistors (OECTs)
- Electrochemical biosensing: The road ahead.
  - Ionogels & OECTs
- Conclusions.

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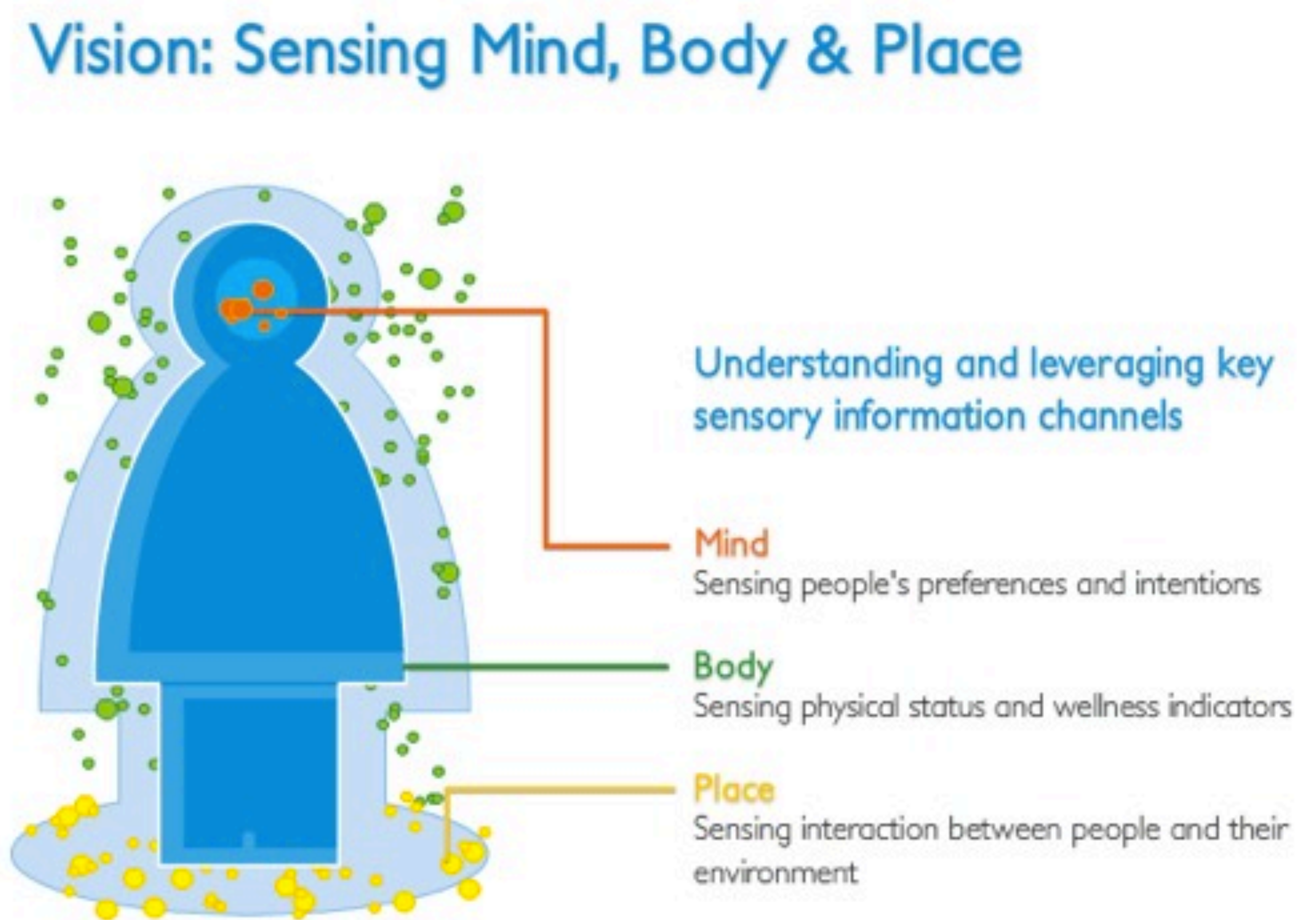

- 5-year, €16.4 million research program to develop next generation Sensor Web Technologies with significant environmental focus
- Brings together fundamental materials science, functional polymers, device prototyping, energy management, adaptive middleware, wearable sensors, distributed environmental monitoring.

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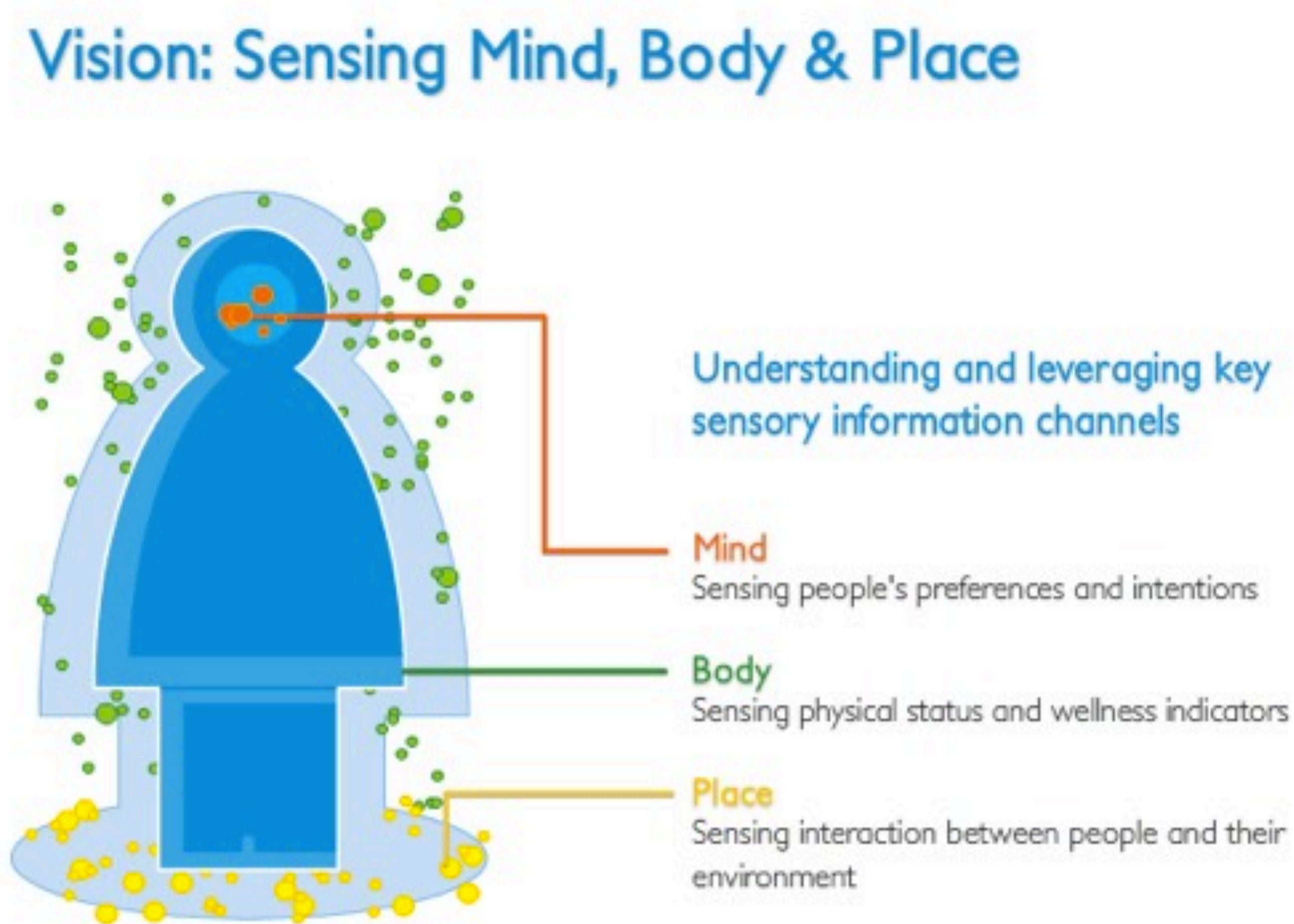

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- Brings together fundamental materials science, functional polymers, device prototyping, energy management, adaptive middleware, wearable sensors, distributed environmental monitoring.

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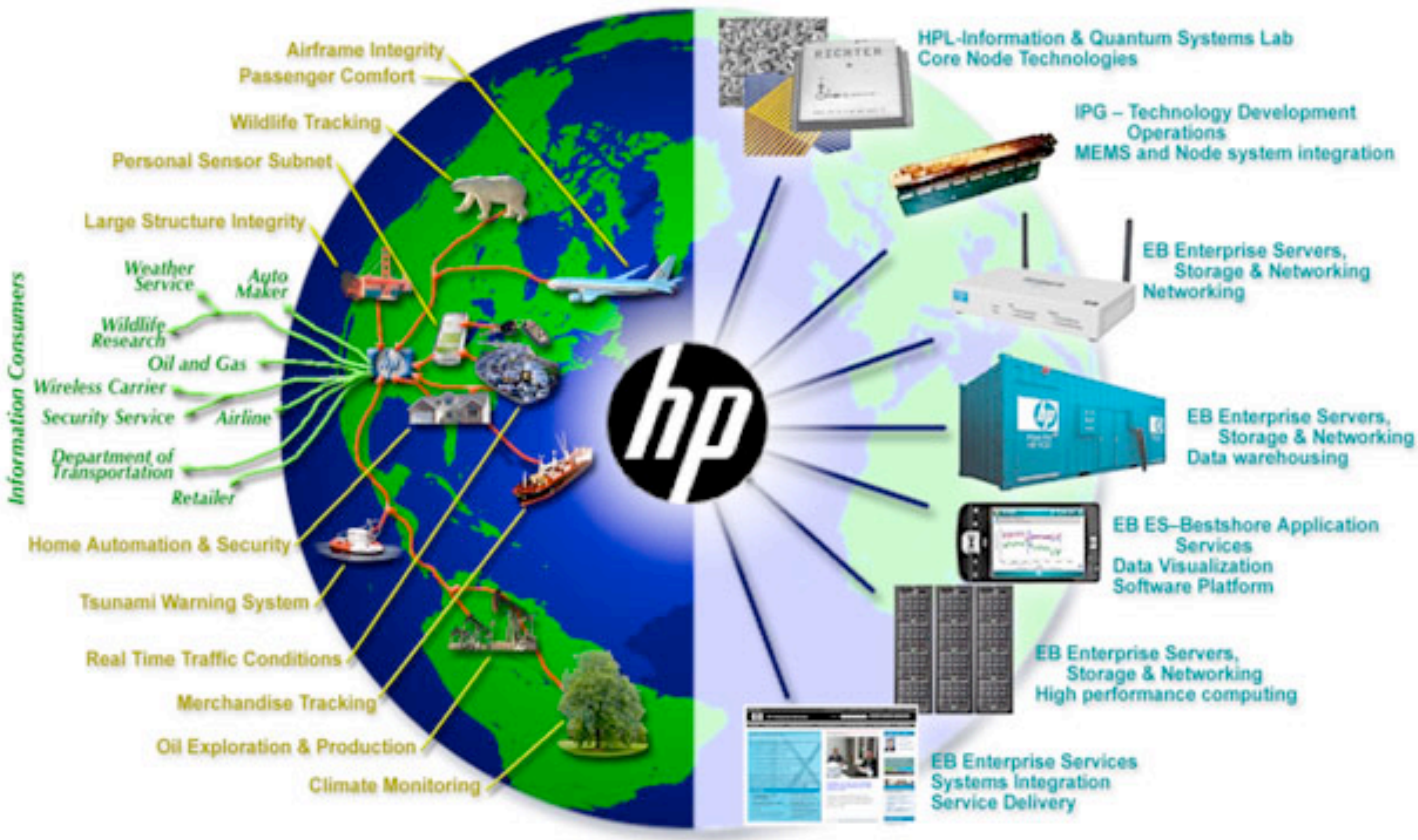
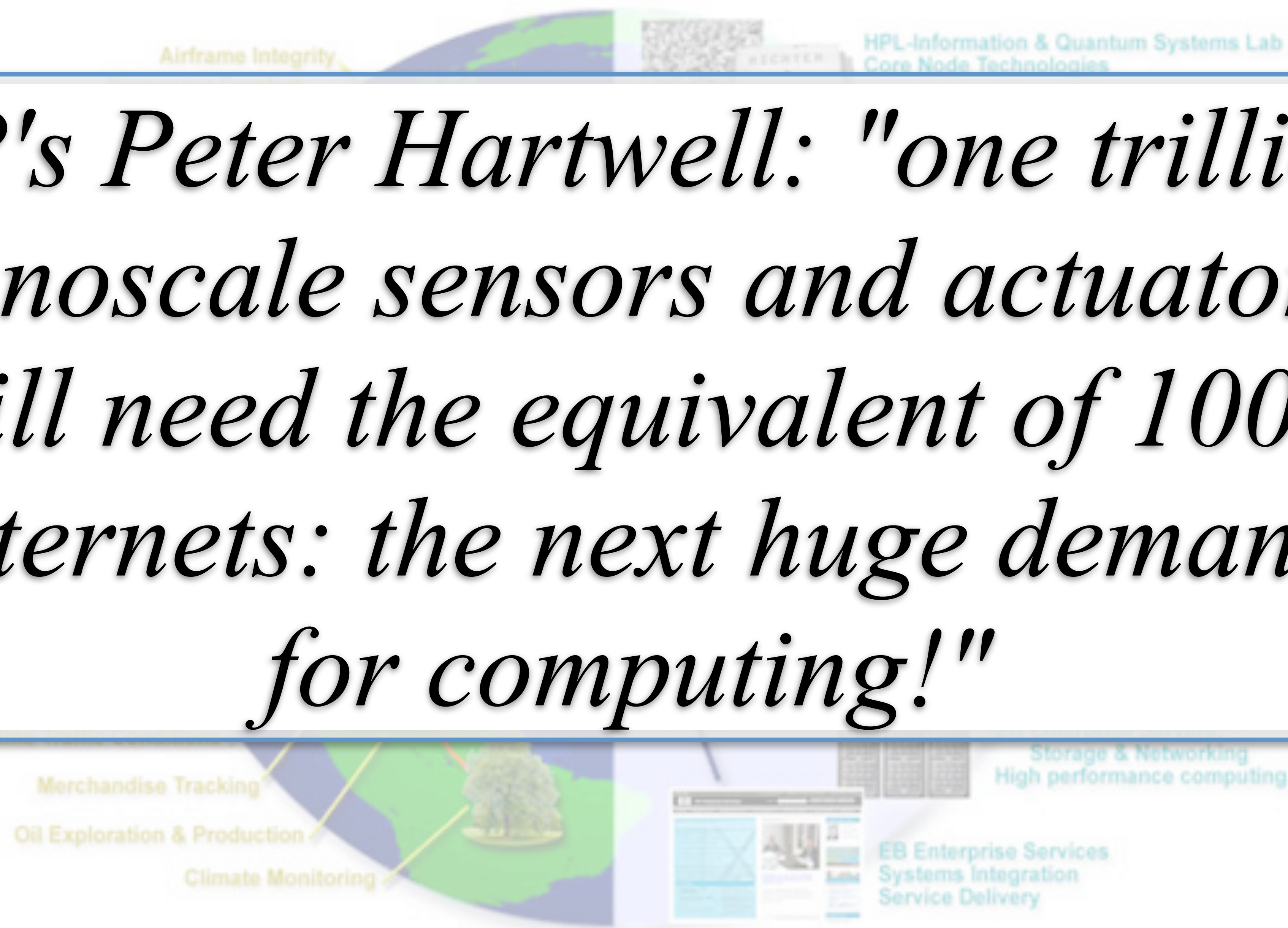
The need for sensors.



http://www.slideshare.net/hewlettpackard/hp-cense-sensor-networks-and-the-pulse-of-the-planet

The need for sensors.



HP's Peter Hartwell: "one trillion nanoscale sensors and actuators will need the equivalent of 1000 internets: the next huge demand for computing!"

http://www.slideshare.net/hewlettpackard/hp-cense-sensor-networks-and-the-pulse-of-the-planet

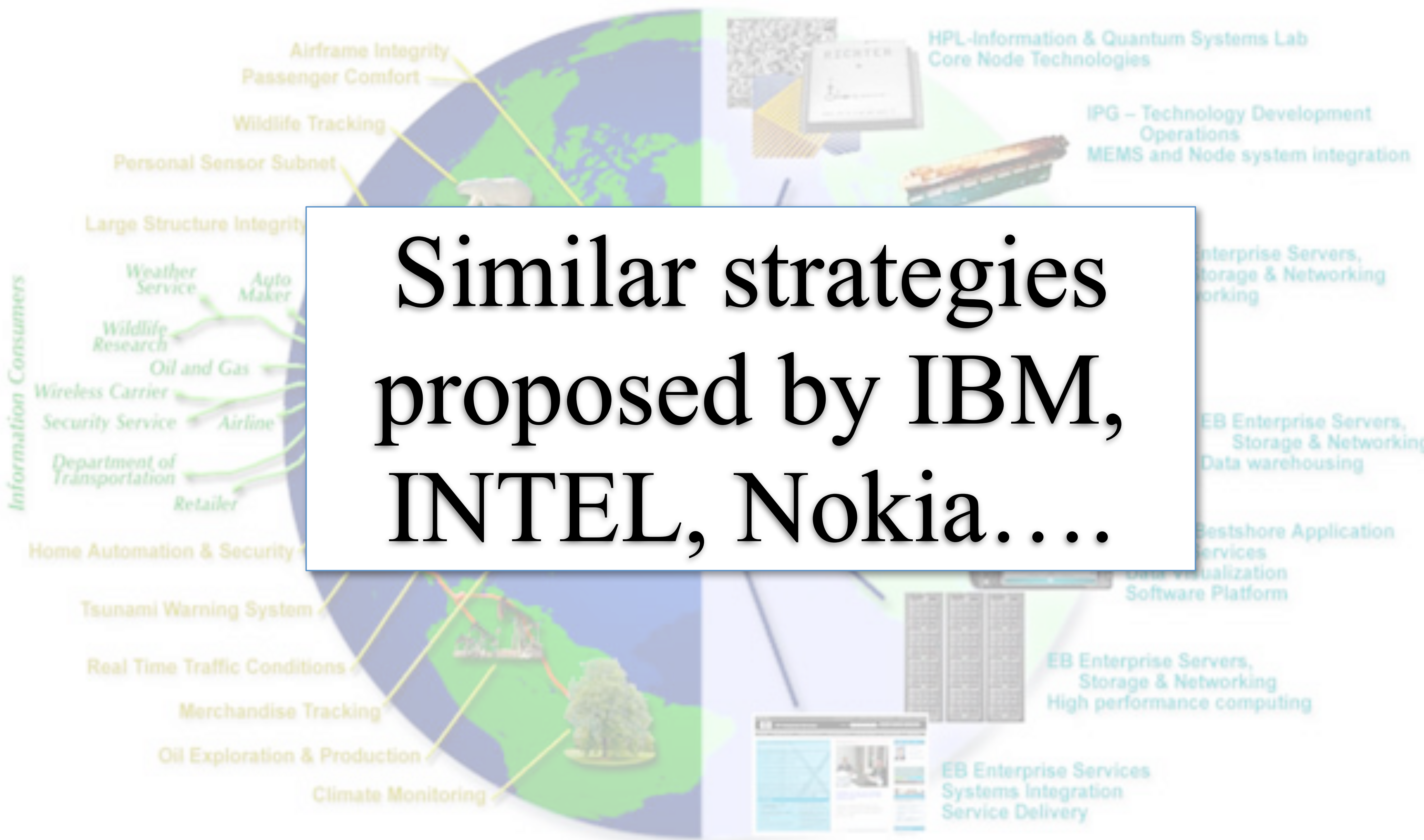
The need for sensors.

Sensing Systems: $ 70 B global market by 2013 (Frost & Sullivan)

Sensing Services: $290 B Global market by 2013 (Harbour research)

http://www.slideshare.net/hewlettpackard/hp-cense-sensor-networks-and-the-pulse-of-the-planet

The need for sensors.



Similar strategies proposed by IBM, INTEL, Nokia….

http://www.slideshare.net/hewlettpackard/hp-cense-sensor-networks-and-the-pulse-of-the-planet

In-house UV Sensor

In-house UV Sensor

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2 hrs Sicily →

← Dublin

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The need for wearable sensors.

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• Wearable sensors allow the continuous monitoring of a person's physiology in a natural setting.

• Health-monitoring systems using electronic textiles are mainly targeting applications based upon physiological parameter measurements, such as body movements or electrocardiography (ECG).

• However, due to their relative complexity, there is very little activity in the development of real-time wearable chemo/bio sensing for sports applications.

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Current Wearable Sensors

PHYSICAL SENSORS

Breath rate, heart rate, activity, posture, skin temperature…

Current Wearable Sensors

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PHYSICAL SENSORS

Breath rate, heart rate, activity, posture, skin temperature…

TRAINTRAK™
heart rate, respiration rate, posture, activity, and GPS location

LIFESHIRT®
Bipolar affective disorder
and schizophrenia
'Activity'

NIKE-APPLE iPOD SPORTS KIT

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Current Wearable Sensors

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Scosche myTrek sends workout vitals to your iPhone, starts shipping now for $130 (video)

By Zach Honig posted Aug 9th 2011 9:40PM

The myTREK utilizes two LEDs combined with a photo sensor to detect minute changes in the user's blood pressure to accurately measure pulse. A built-in accelerometer allows the myTREK to adjust for movement during exercise from the user's heartbeat allowing for an extremely accurate measurement of pulse and calories burned.

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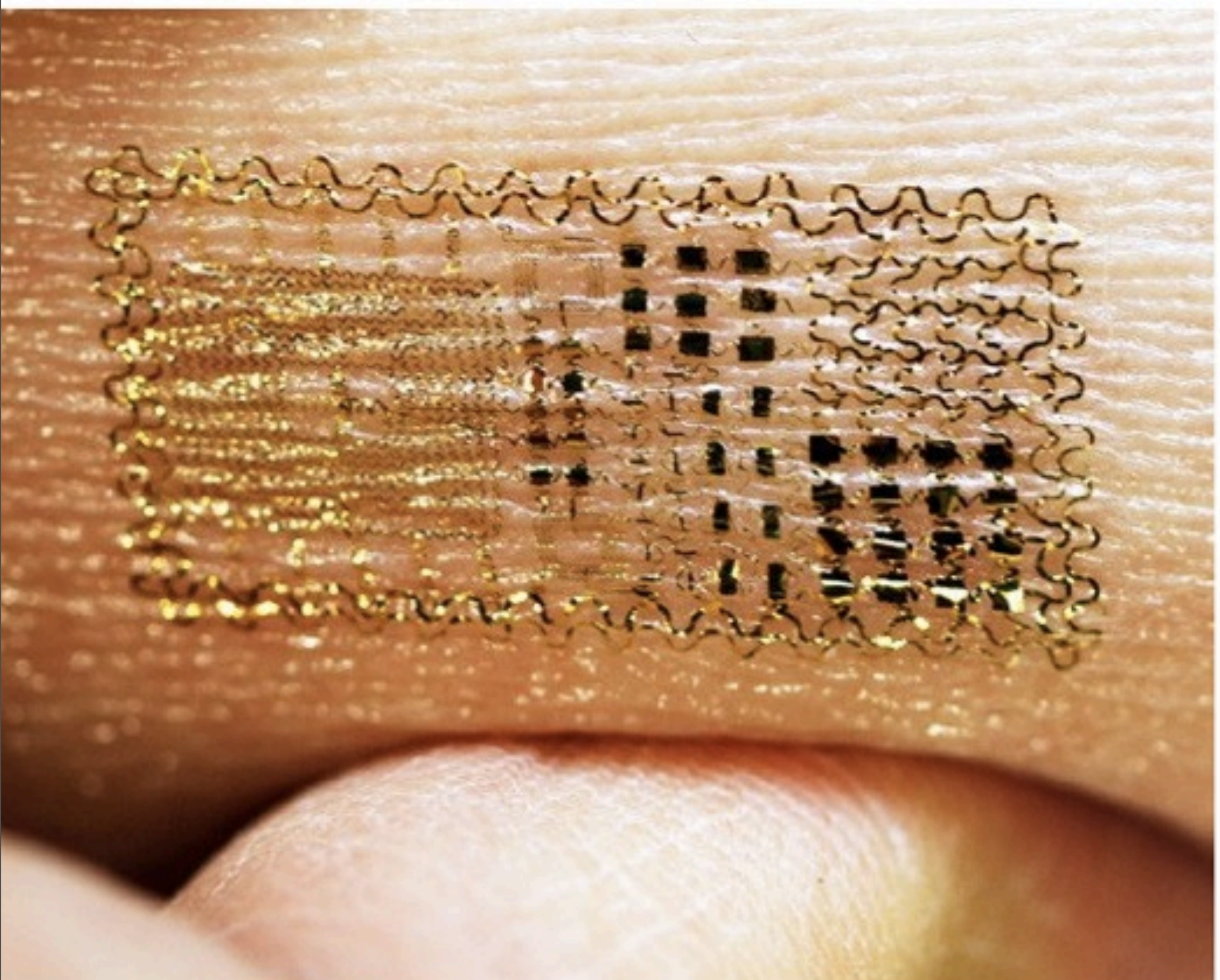
Current Wearable Sensors



John Rogers @ University of Illinois

11/08/2011

Ultra-thin, self-adhesive electronics device that can effectively measure data about the human heart, brain waves and muscle activity--all without the use of bulky equipment, conductive fluids or glues.

Link :http://www.nsf.gov/news/news_summ.jsp?cntn_id=121343&org=NSF&from=news

Current Wearable Sensors

You are here: Home > Health > Health News

The 'electronic skin' patches that can tell when you're ill

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Tuesday March 27 2012

Electronic skin patches that monitor a patient's health while at home are being developed by scientists.

The first patches, aimed at athletes, are expected to be available for commercial sale later th... Getty Images

American researchers say the tiny patches, with tattoo-like sensors that wirelessly diagnose and treat health problems, can act as a person's own internal doctor.

The "electronic skin" patches, about as a thick as a human hair, check a patient's "vital signs" and transmits the data to a computer or mobile phone.

Scientists behind the project also say they could be used by healthy people to detect the early signs of illness.

The first patches, aimed at athletes, are expected to be available for commercial sale later this year.

The information then gets sent to the person's doctor for further analysis, who can then act on any worrying conclusions.

They are made of a silicon membrane that stretches and moves with the body.

Sensors contained within the patches can measure heart rates and temperatures and monitor whether injured muscles are healing.

Also in Health

- It's official! C... help keep yo...
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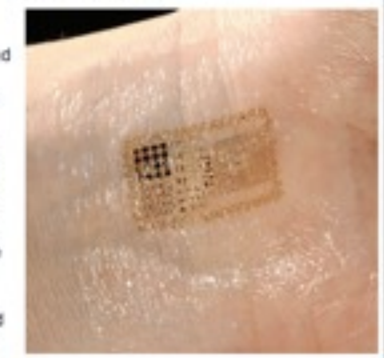
Temporary tattoo to give you the sporting edge

27 March 2012

Hydration is incredibly important. In sport dehydration can affect your performance and decision making abilities but by the time you get thirsty you're already dehydrated. This Saturday, Nascar racer Paulie Harraka will be using a device based on John Rogers work at the University of Illinois Urbana Champaign to monitor his hydration levels as he races.

Rogers' device is an extension of his work presented in his *Science* paper last year, and consists of a 'temporary tattoo' of flexible electronics in direct contact with the skin. He gave the update on his work to delegates at the American Chemical Society annual meeting in San Diego, US.



Rogers' electronic tattoos could one day be used to control prosthetic limbs

© John Rogers

Silicon electronics are extremely useful for a huge range of uses, but are rigid and inflexible, unlike the human body. Previously, Rogers has shown that nanometre thin layers of silicon have much better mechanical properties. Forming these into snakelike bends makes circuits that not only flex and bend, but also stretch, and at such sizes the circuits can then stick onto the body using van der Waals interactions.

Roger's circuits can be peeled from their backing and put in place using just water. They can be protected by coating them with a modified form of over-the-counter spray-on plasters or bandages, which keeps them safe from sweating, wear and tear and even being washed with soapy water.

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Chemistry World shared a link.

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www.rsc.org

Hydration is incredibly important. In sport dehydration can affect your performance and decision making abilities but by the time you get thirsty you're already dehydrated. This Saturday, Nascar racer Paulie Harraka will be using a device based on John Rogers work at the University of Illinois Urban...

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Link : ... =news

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Current Wearable Sensors

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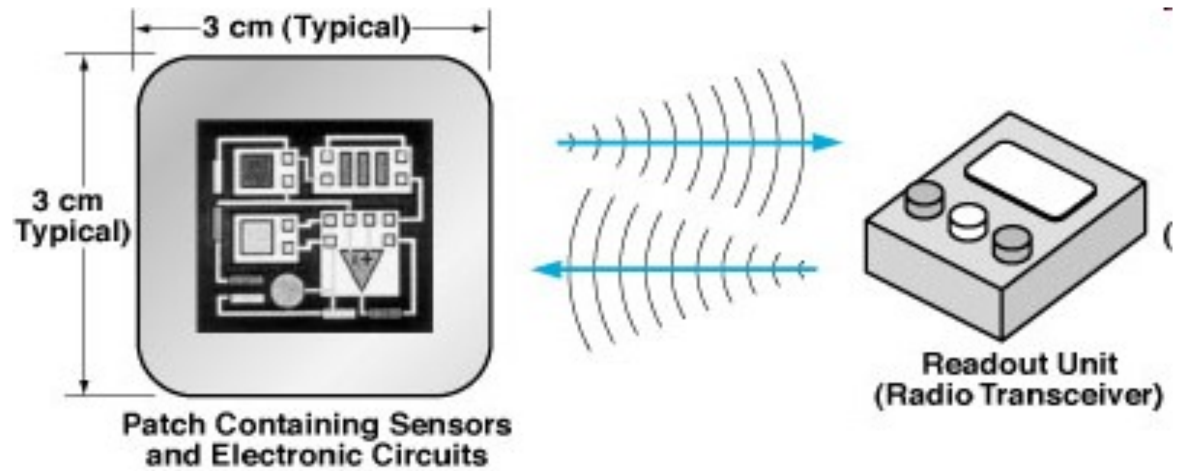
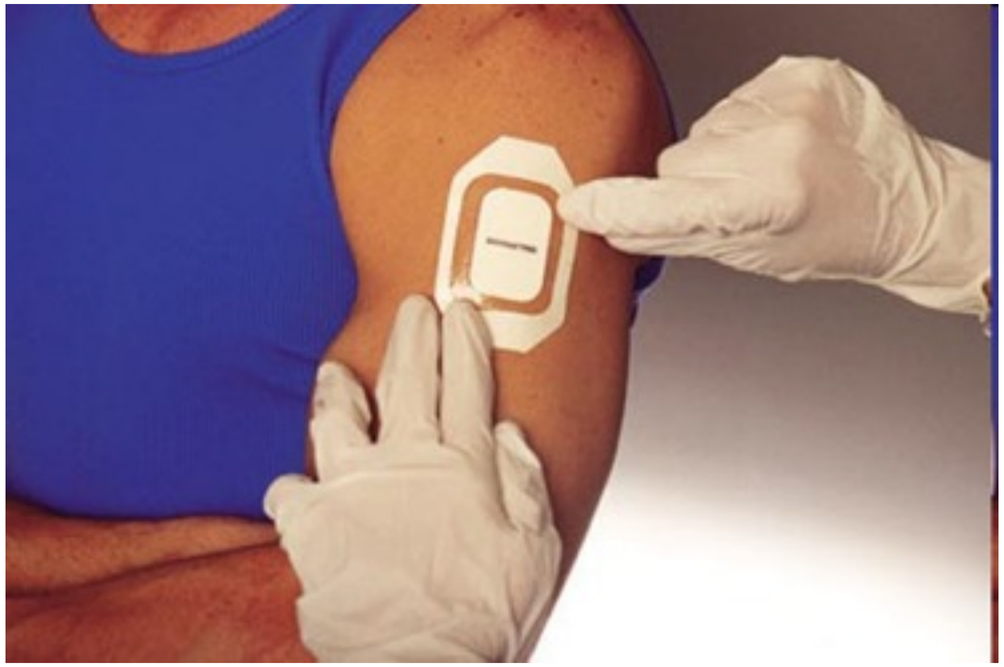
Glucowatch



NASA: Wearable sensor patches

Abbot Freestyle Navigator



PharmChek Sweat Patch

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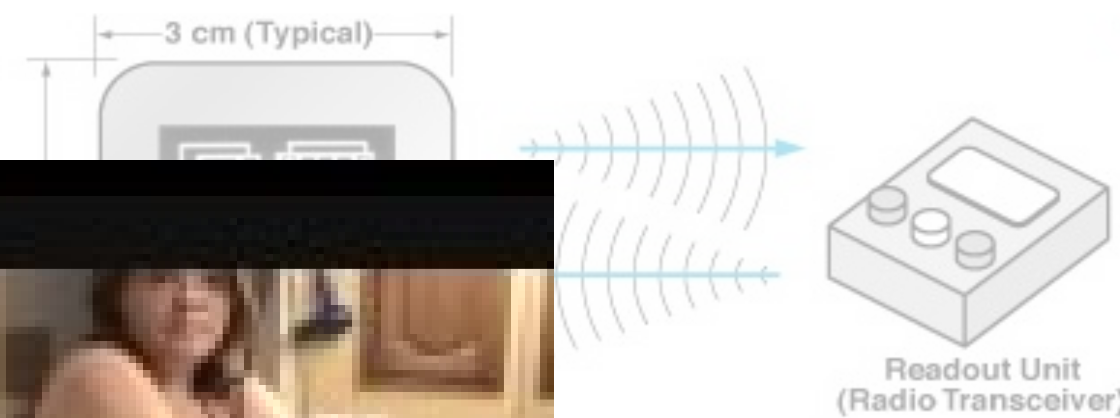
Current Wearable Sensors

Glucowatch



le sensor patches

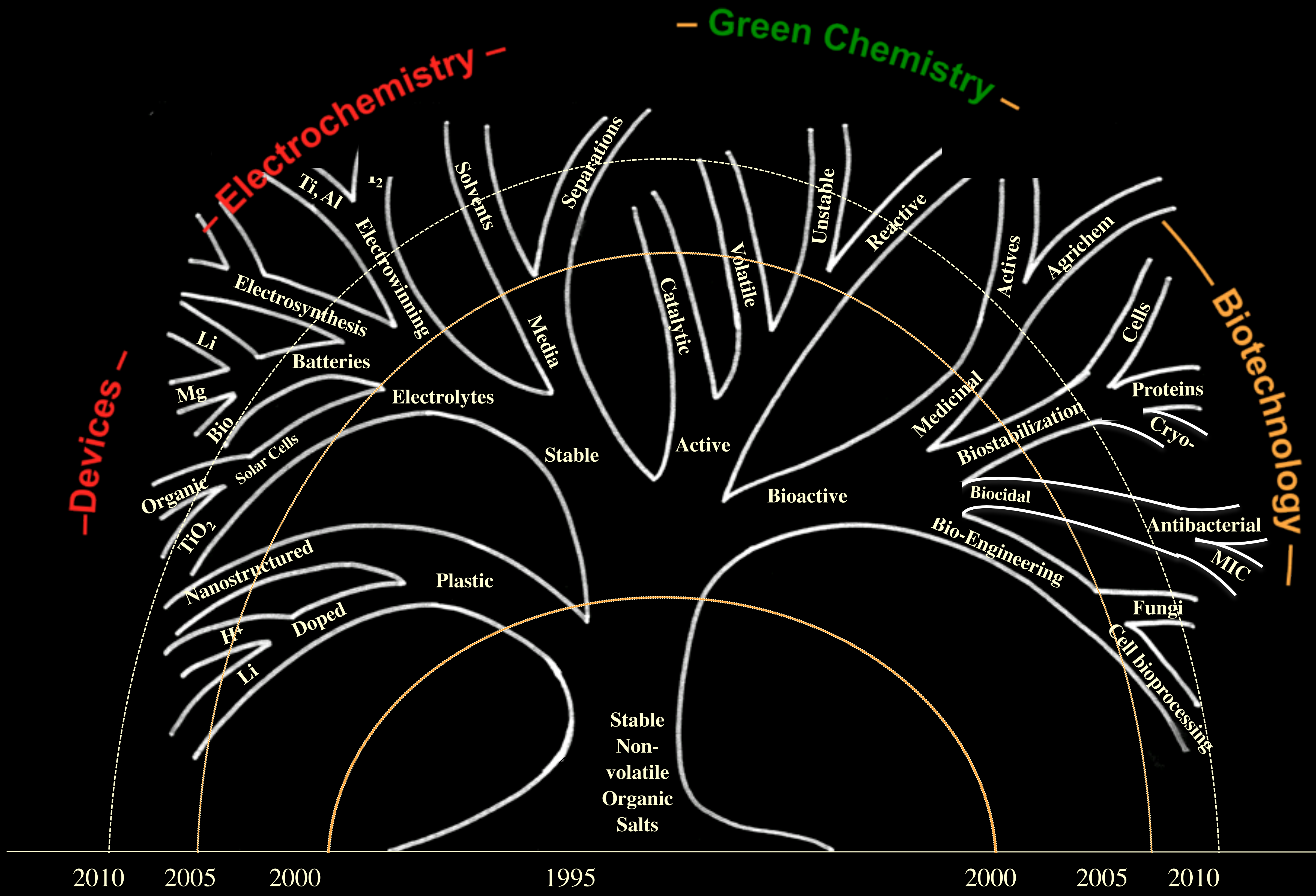
SAMPLING BIG ISSUE!!!!

http://www.youtube.com/watch?v=pmMo3AYKOk0

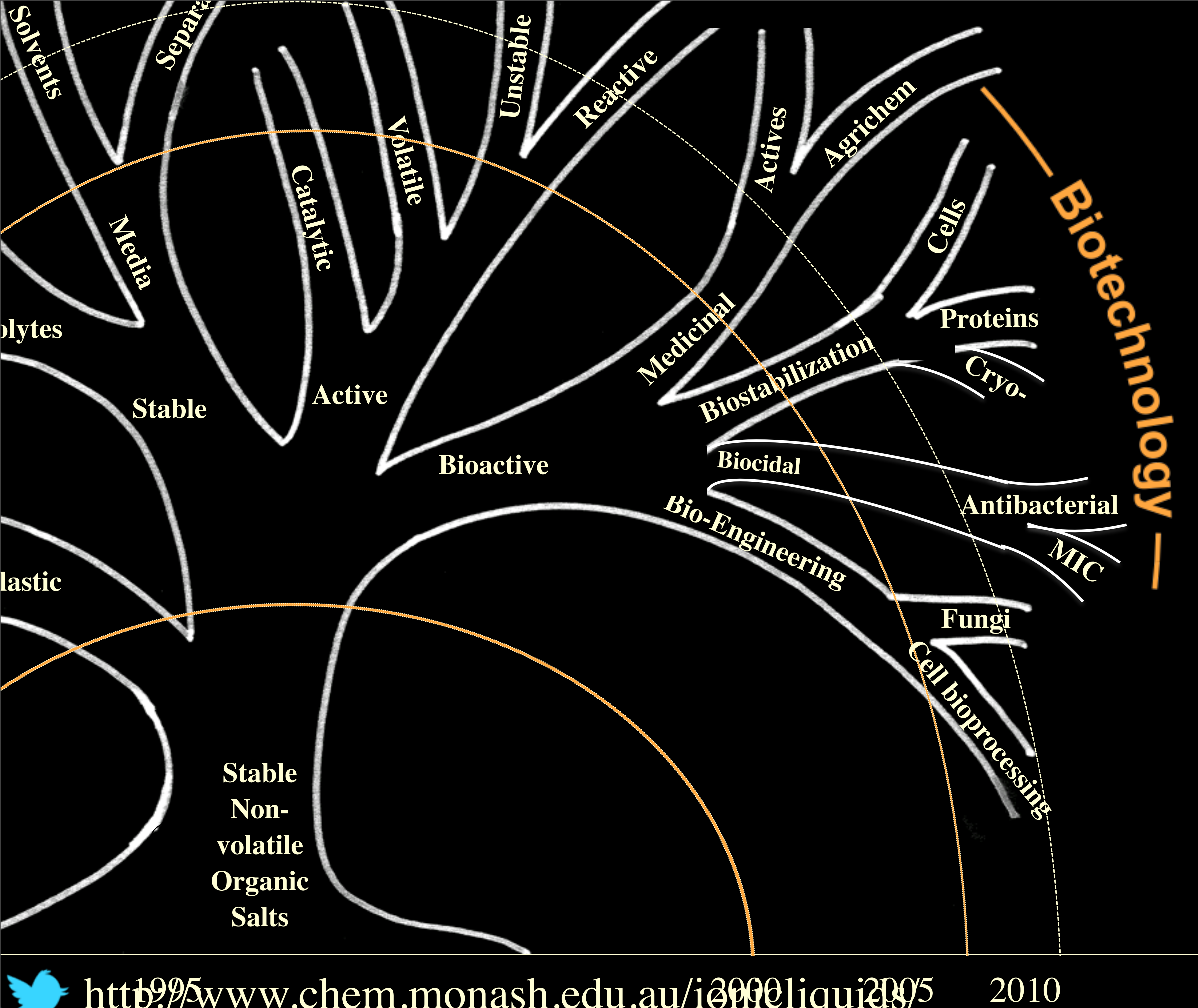
Abbot Freestyle Navigator

PharmChek Sweat Patch

Ideas Tree for Ionic liquids



http://www.chem.monash.edu.au/ionicliquids/



http://www.chem.monash.edu.au/ionicliquids/

Ionic Liquids: A brief introduction

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- Ionic liquids (ILs) have evolved as a new type of non-aqueous solvents for biocatalysis, mainly due to their unique and tunable physical properties [1]

Factors that affect Enzyme activity in ILs

IL polarity

Hydrogen bonding bascicity

Viscosity

Ion kosmotropicity

Enzyme dissolution

[1] H. Zhao, *J. Chem. Tech. Biotech*, 2010, **85**, 891-907.

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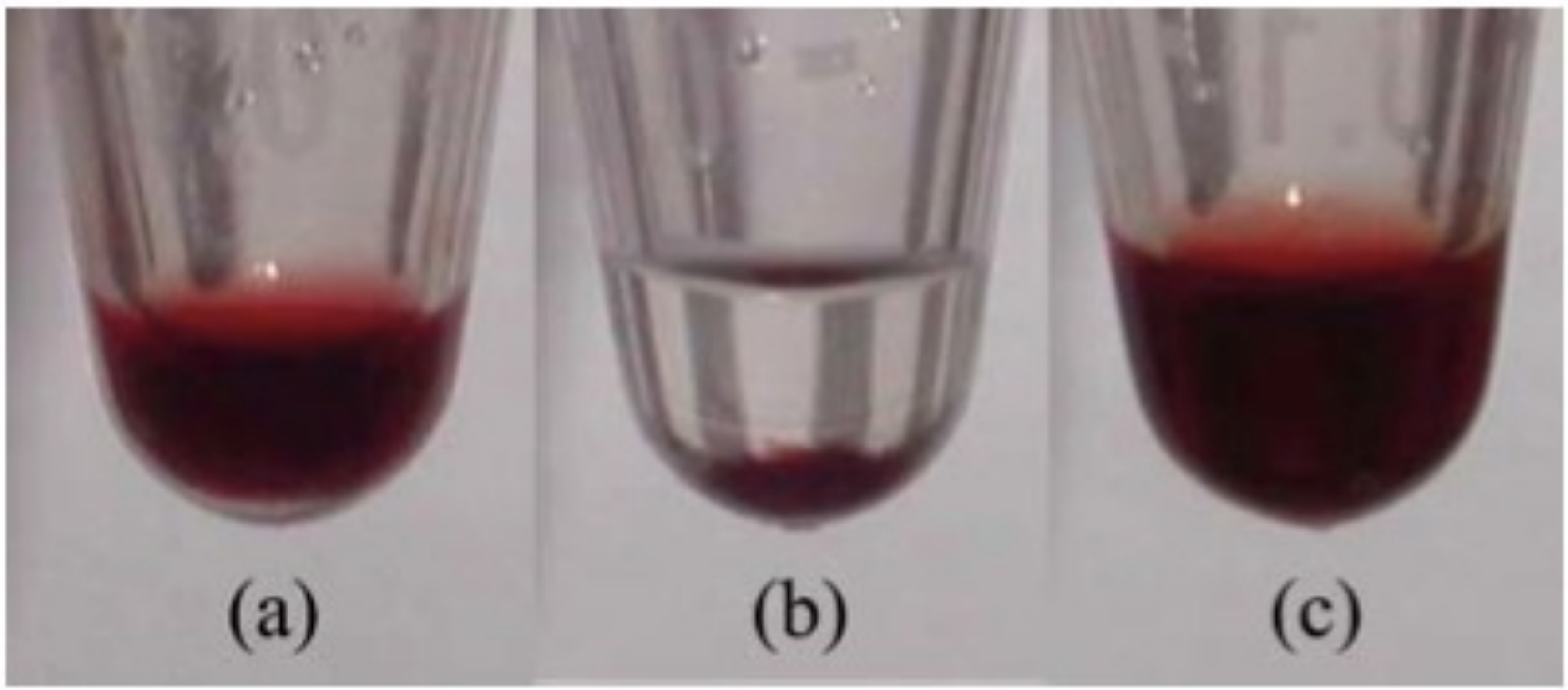
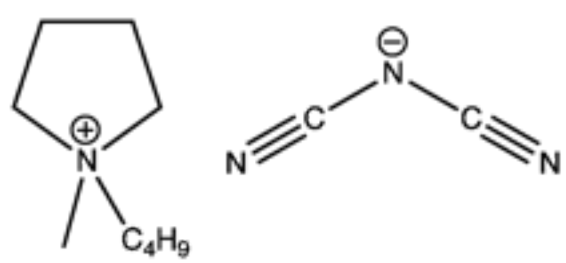
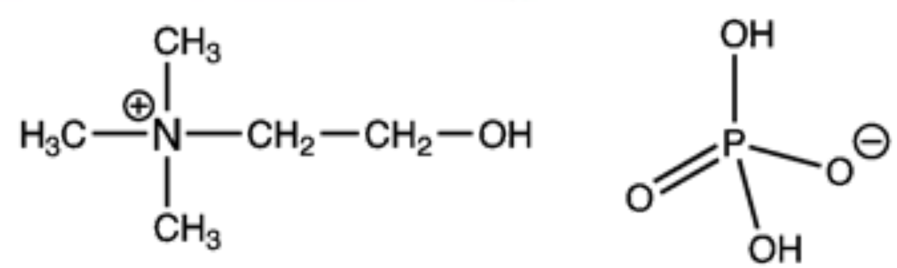
Ionic Liquids: A brief introduction

Through smart design enzyme stability can be greatly enhanced



PBS





[2] K. Fujita, D. R. MacFarlane and M. Forsyth, *Chem. Commun.*, 2005, 4804-4806.

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Ionic Liquids: A brief introduction

Through smart design enzyme stability can be greatly enhanced

- Enhanced solubility of cytochrome c.

- dhp anion provided both a proton activity similar to that in neutral water as well as hydrogen bonding donor and acceptor sites.

- Choline DHP showed enzyme stability up to 130 oC [2]

[2] K. Fujita, D. R. MacFarlane and M. Forsyth, *Chem. Commun.*, 2005, 4804-4806.

Sensing Platform: Ionic liquids

- Point-of-care (POC) glucose biosensors play an important role in the management of blood sugar levels in patients with diabetes.

- One of the most commonly used enzymes in glucose biosensors is Glucose Oxidase (GOx).

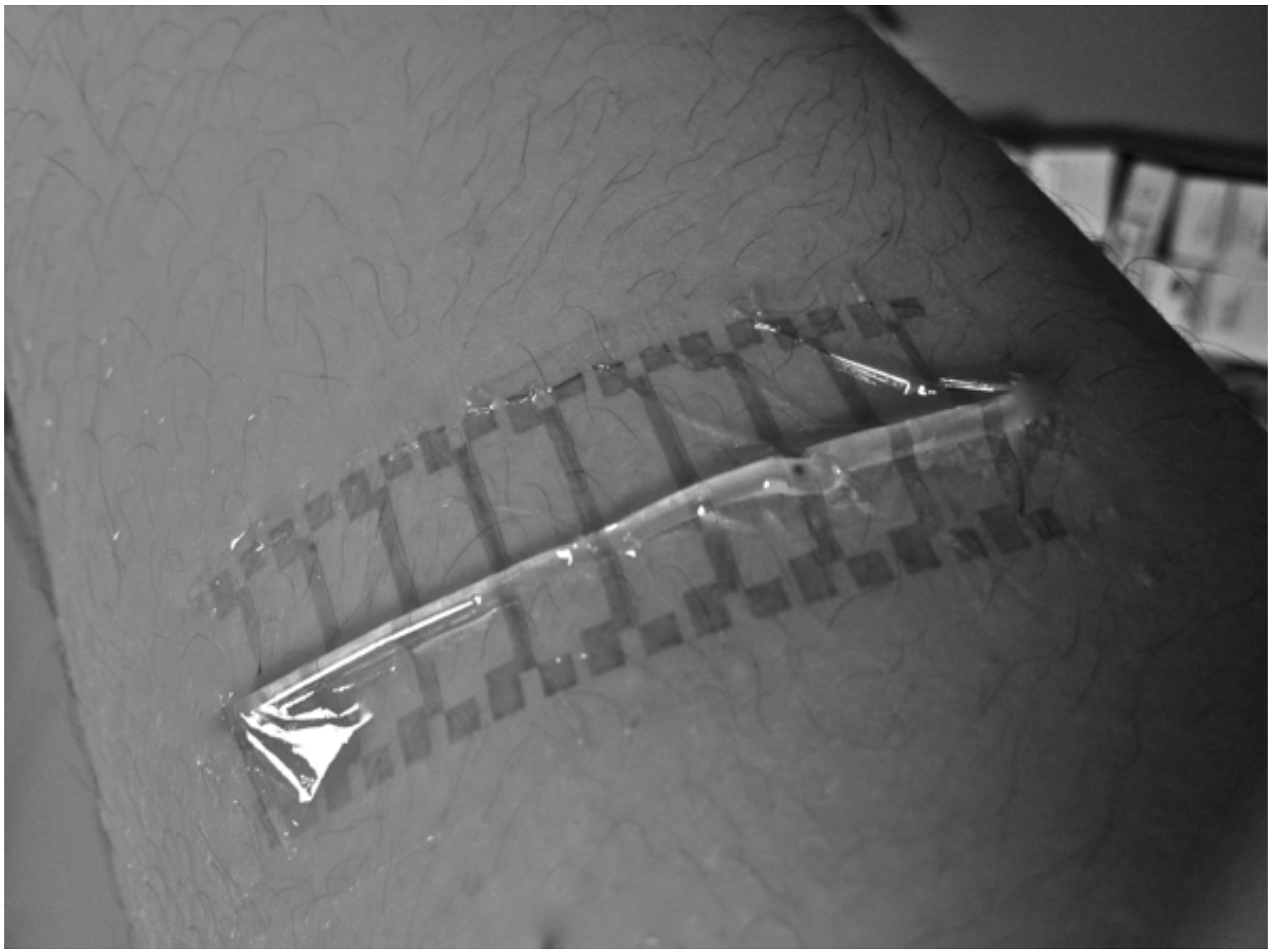
- Amperometric biosensors employing IL's have been reported previously, for example, ([C_4mIm][BF_4]) has been used as a mediator in a electrochemical H_2O_2 biosensor[3].

- This interest is driven by the need to find molecular environments in which enzymes are highly stabilized while retaining redox activity.

[3] Y. Liu, M. Wang, J. Li, Z. Li, P. He, H. Liu and J. Li, *Chem. Commun.*, 2005, 1778-1780.

Sensing Platform: OECTs

Organic Electrochemical Transistors (OECTs)



Sensing Platform: OECTs

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• The field of organic electronics has grown significantly in the past 20 years largely due to the many desirable properties of organic semiconductors such as low cost, ease of processing and tunability through synthetic chemistry.

• A prototypical semiconductor used in OECTs is poly(3,4-ethylenedioxythiophene) doped with poly(styrenesulfonate) (PEDOT:PSS), a material that is commercially available and stable under a variety of conditions.

• PEDOT:PSS is a degenerately doped p-type semiconductor (commonly referred to as a conducting polymer), where holes on the PEDOT are compensated by acceptors (SO^{3-}) on the PSS.

• OECTs have been utilized in a variety of biosensing applications such as the detection of metabolites, ions, neurotransmitters, cells, antibodies and DNA

[4] D. Bernards, G. Malliaras, "steady state and transient response of organic electrochemical transistors" Adv. Func Mat 2007.

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Sensing Platform: OECTs

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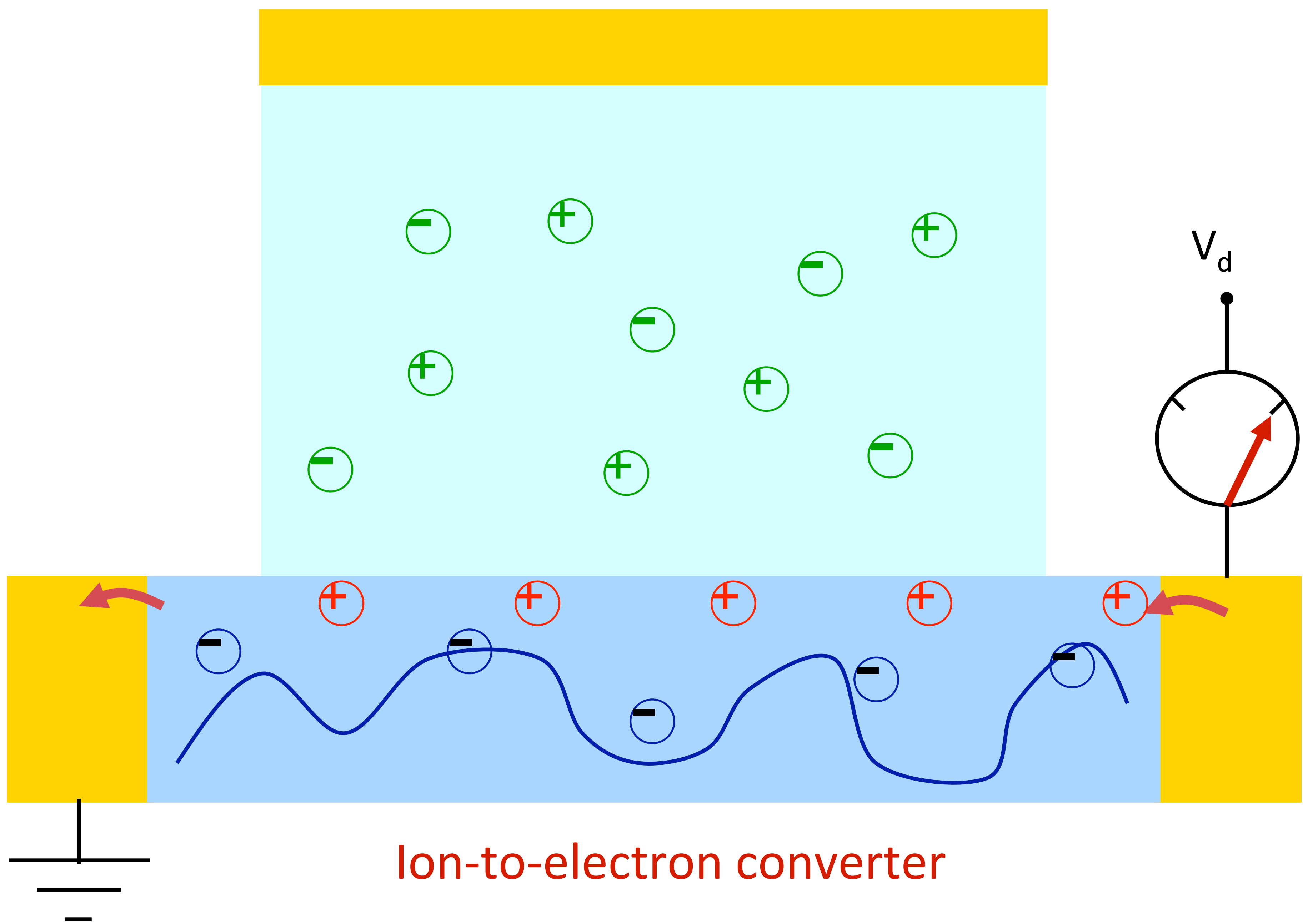
The organic electrochemical transistor [4]



Ion-to-electron converter

[4] D. Bernards, G. Malliaras, "steady state and transient response of organic electrochemical transistors" Adv. Func Mat 2007.

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Sensing Platform: OECTs

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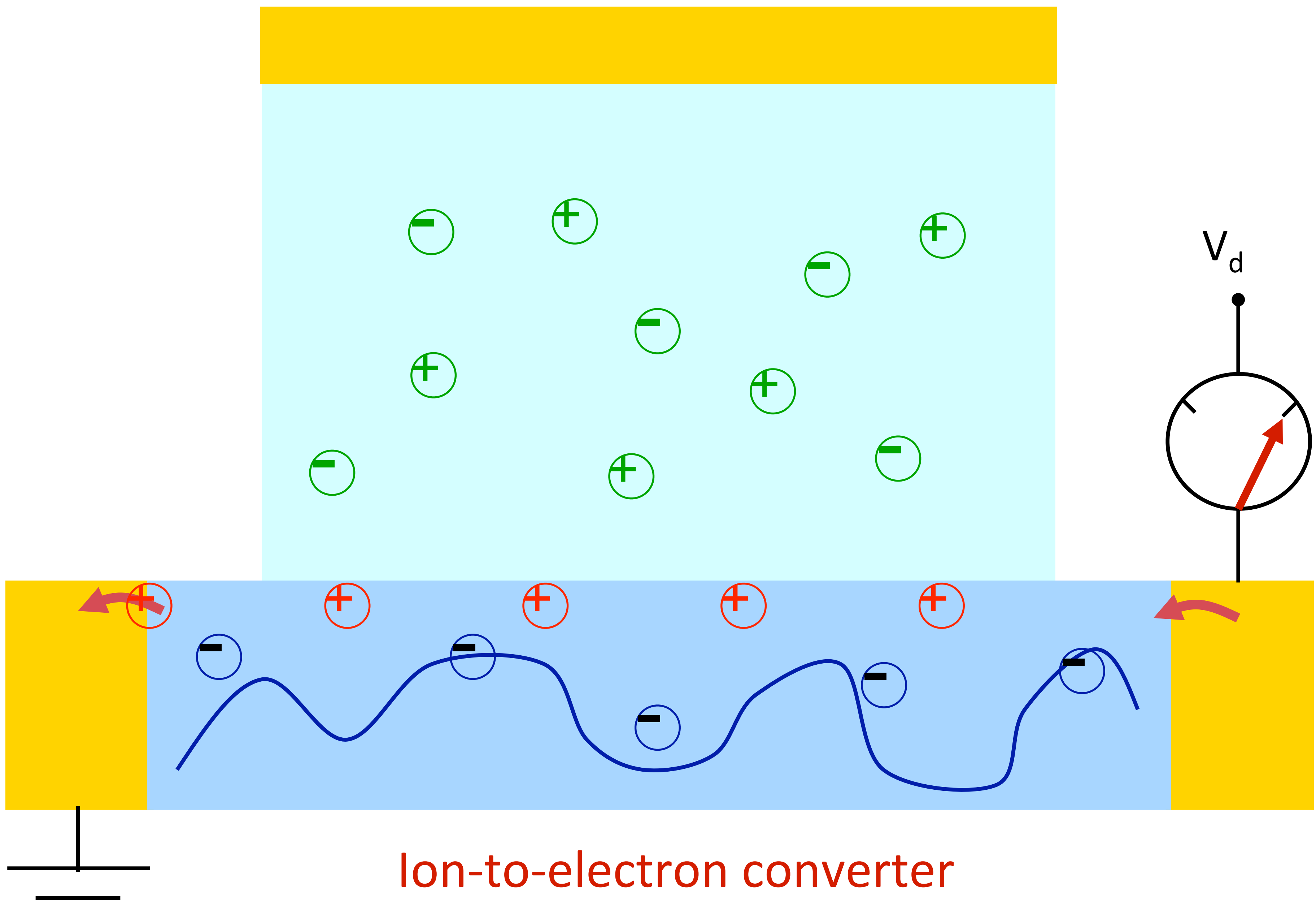
The organic electrochemical transistor [4]



[4] D. Bernards, G. Malliaras, "steady state and transient response of organic electrochemical transistors" Adv. Func Mat 2007.

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Sensing Platform: OECTs

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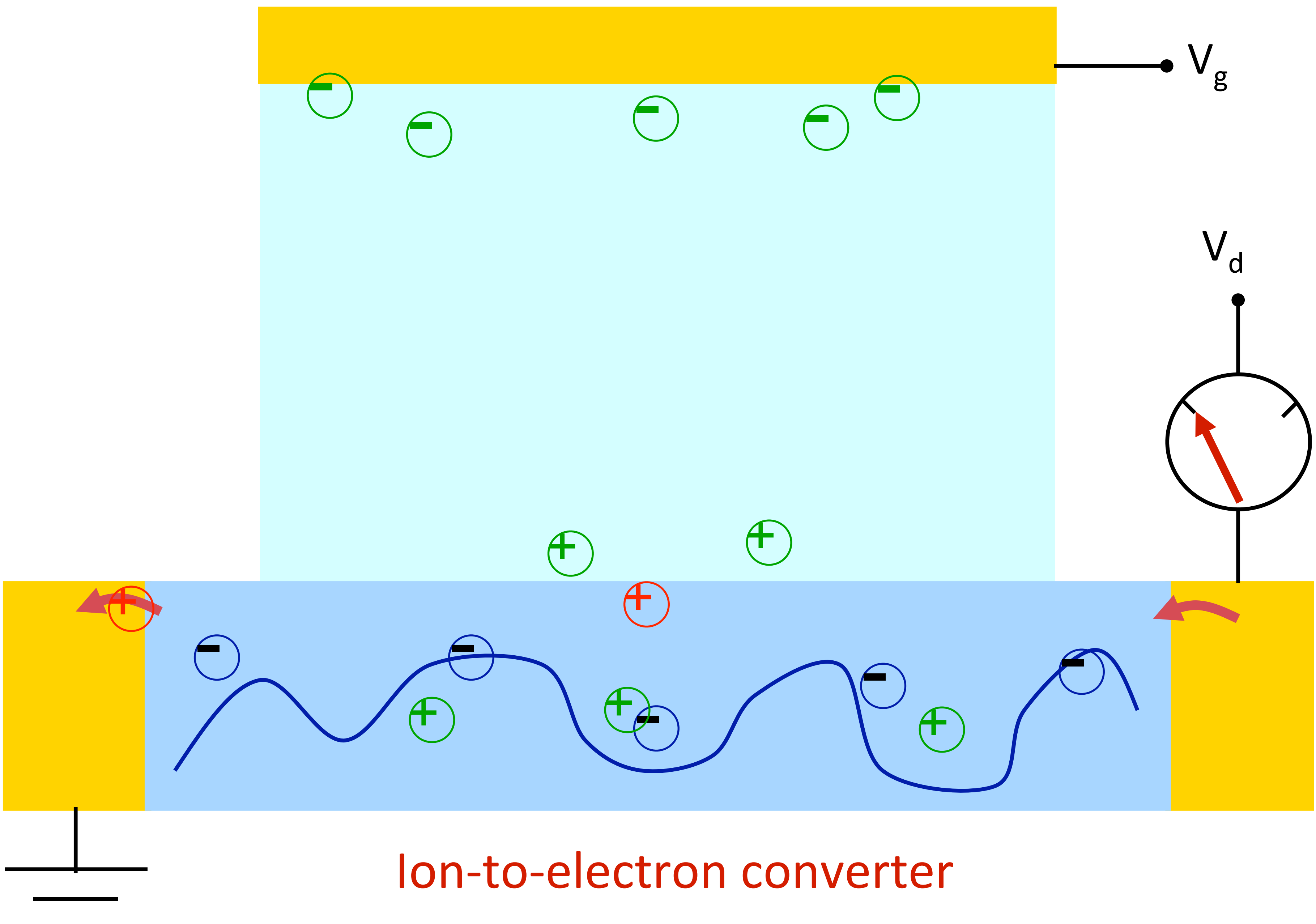
The organic electrochemical transistor [4]



Ion-to-electron converter

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Sensing Platform: OECTs

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• Response of the transistor is defined as the difference in modulation level of the drain channel during application of a gate voltage in the absence and presence of a target analyte

The Aim:

• To develop an enzymatic sensor based on an OECT that uses an IL as an integral part of its structure.

• The strategy involves patterning the RTIL over the active area of the OECT, and using it as a reservoir for the enzyme and the mediator.

• GOx enzyme for glucose detection.

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Sensing Platform: Ionic liquids & OECTs

- Important properties of the electrolyte for this device must include wetting the PEDOT : PSS film.

[5] S. Y. Yang, F. Cicoira, R. Byrne, F. Benito-Lopez, D. Diamond, R. A. Owens and G. G. Malliaras, *Chem. Commun.*, 2010, **46**, 7972-7974.

Sensing Platform: Ionic liquids & OECTs

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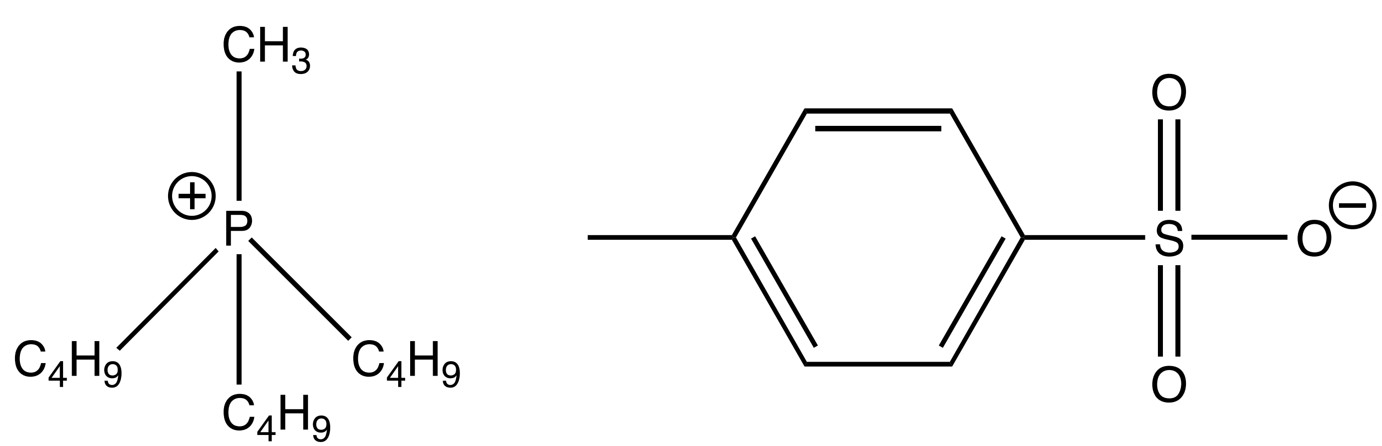
- Important properties of the electrolyte for this device must include wetting the PEDOT : PSS film.

- This allows the enzyme and the mediator to be patterned over the active area of the device.

- The IL should be miscible with the aqueous phase (PBS).

- Triisobutyl(methyl)phosphonium Tosylate ($[P_{1,4,4,4}][Tos]$) due to the hydrophilic nature of the cation / anion.



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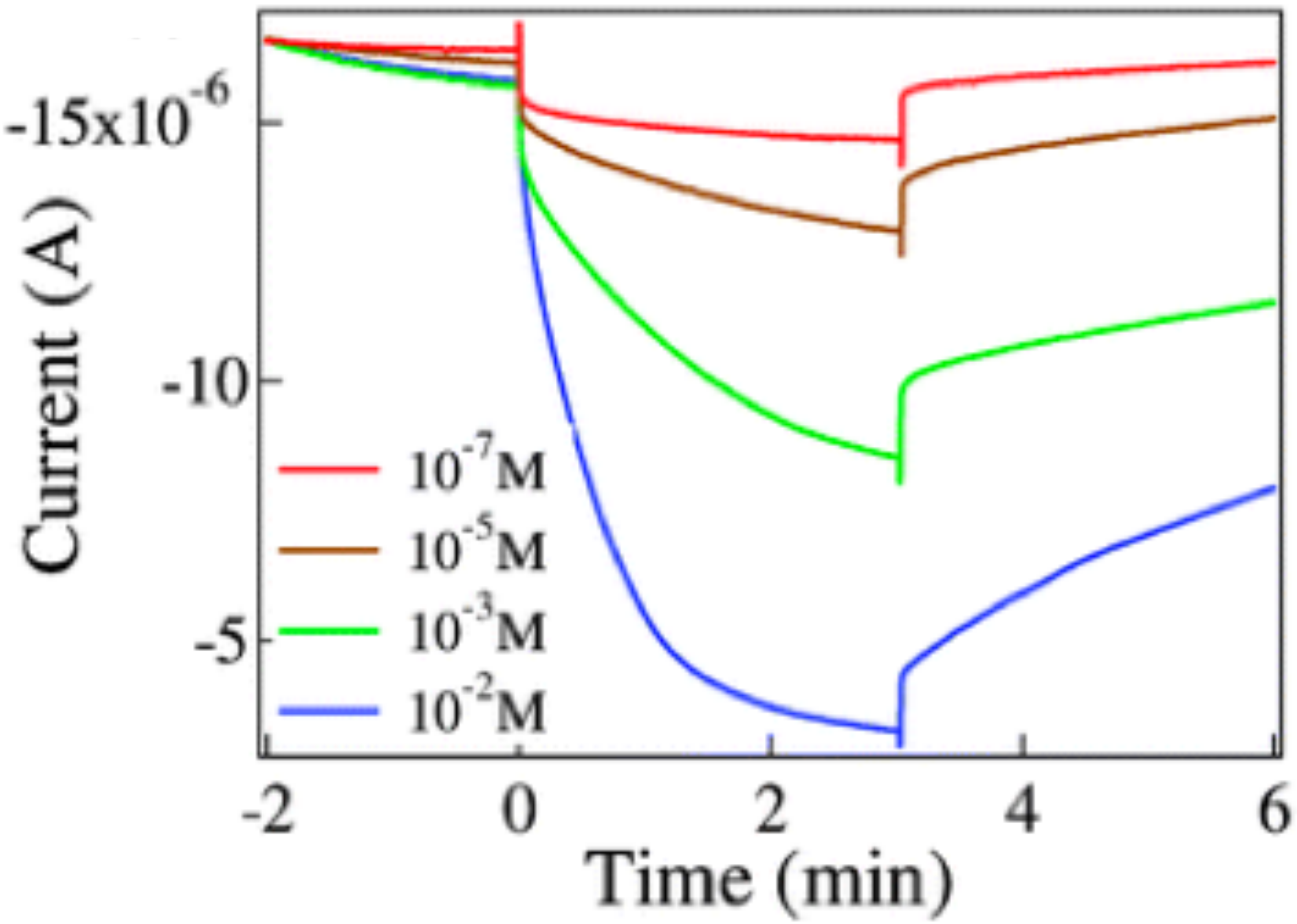
Sensing Platform: Ionic liquids & OECTs

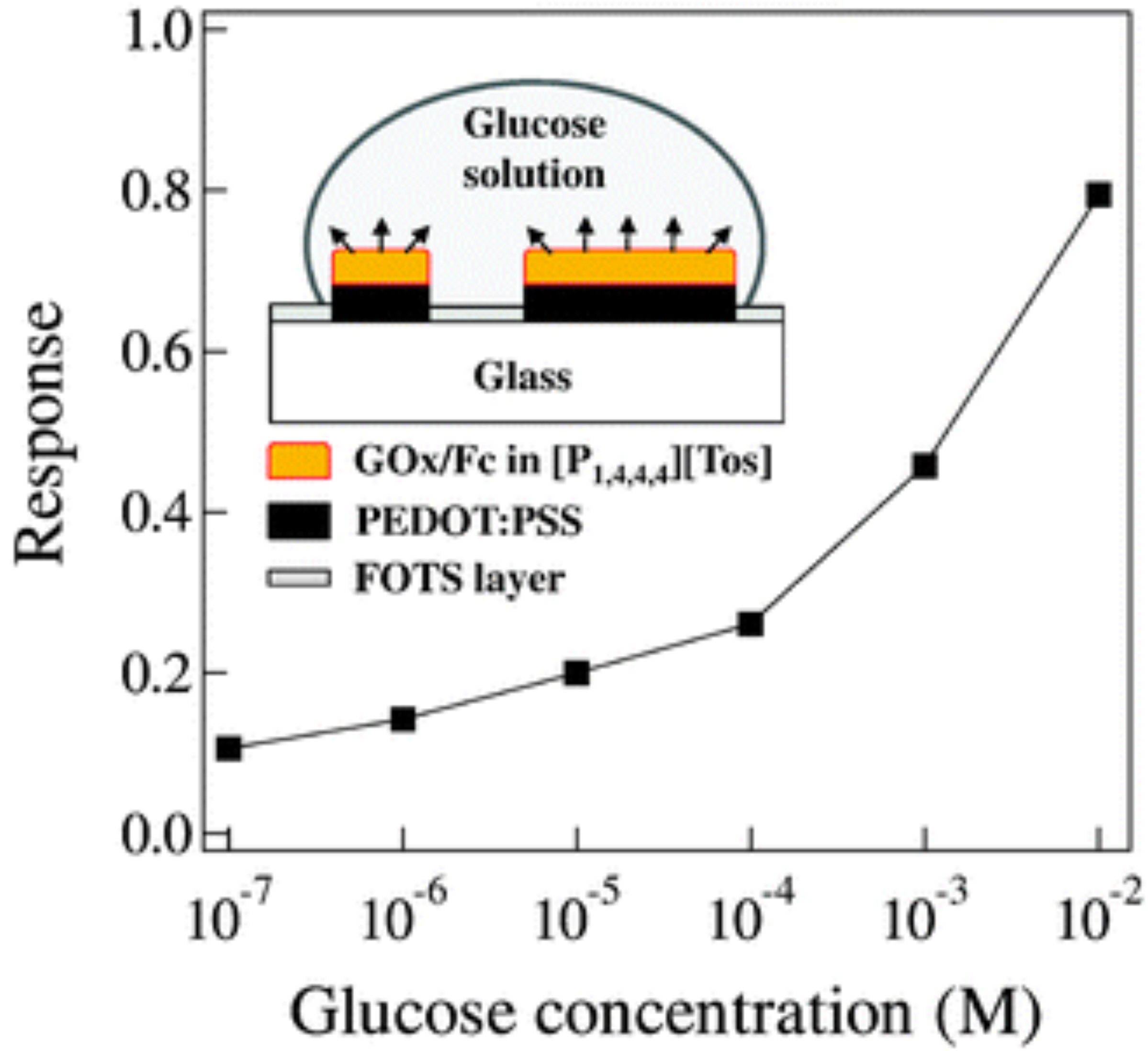


- The transient response of the drain current of an OECT upon application of a gate voltage of 0.4 V and duration of 3 min. The drain voltage was -0.2 V.

[5] S. Y. Yang, F. Cicoira, R. Byrne, F. Benito-Lopez, D. Diamond, R. A. Owens and G. G. Malliaras, *Chem. Commun.*, 2010, **46**, 7972-7974.

Sensing Platform: Ionic liquids & OECTs

- Current modulation of the OECT as a function of glucose concentration.



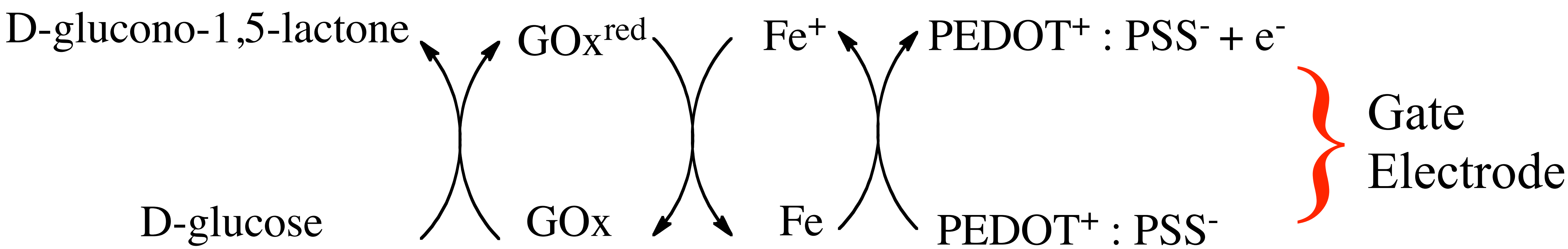
- Inset shows the concept of device operation, and the arrows indicate the dissolution of the RTIL carrying the enzyme and the mediator into the analyte solution.

[5] S. Y. Yang, F. Cicoira, R. Byrne, F. Benito-Lopez, D. Diamond, R. A. Owens and G. G. Malliaras, *Chem. Commun.*, 2010, **46**, 7972-7974.

Sensing Platform: Ionic liquids & OECTs

• The data show the characteristic decrease of drain current upon gating which has been understood on the basis of the reactions shown below

(A)



(B) $PEDOT^{+} : PSS^{-} + M^{+} + e^{-} \longrightarrow PEDOT + M^{+}: PSS^{-}$ } Drain Electrode

Reactions at the gate electrode **(a)** and at the channel **(b)** of the OECT.

Sensing Platform: Ionic liquids & OECTs

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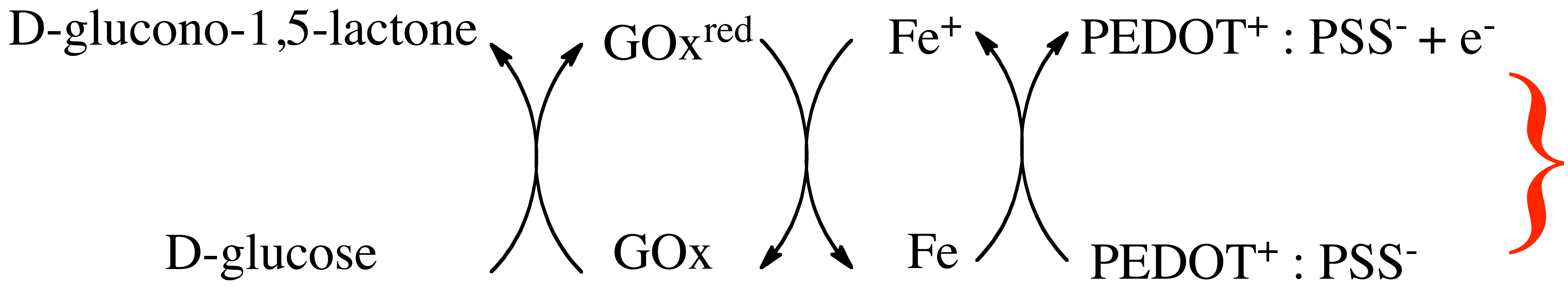
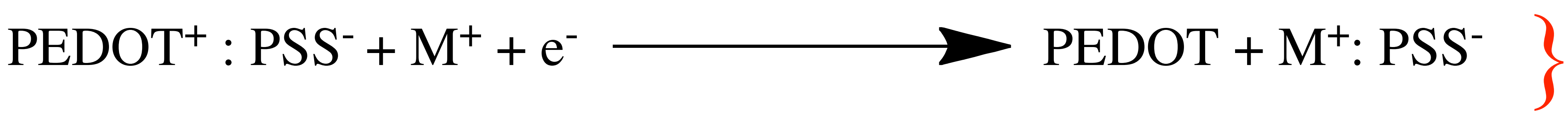
(A)



Gate Electrode

(B)



Drain Electrode

- As glucose in the solution is oxidised, the enzyme (GOx) itself is reduced, and cycles back with the help of the Fc/ferricenium ion (Fc+) couple, which shuttles electrons to the gate electrode **(A)**.

- For example, for 10^{-2} M of glucose, this cascade of reactions causes a current of 8×10^{-8} A to flow to the gate electrode.

- At the same time, cations from the solution (M+) enter the PEDOT : PSS channel and dedope it. **(B)**

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Sensing Platform: Ionic liquids & OECTs

Conclusions:

- Successful integration of an OECT with an IL as electrolyte.

Sensing Platform: Ionic liquids & OECTs

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Conclusions:

- Successful integration of an OECT with an IL as electrolyte.

- The ionic liquid was confined on the surface of the transistor using a photolithographically patterned hydrophobic monolayer.

- The enzyme was in a dispersed state in the ionic liquid, which may prove to be a good strategy for improving long-term storage.

- Using the glucose/ glucose oxidase pair as a model, it was demonstrated the analyte detection in the 10^{-7} to 10^{-2} M concentration range.

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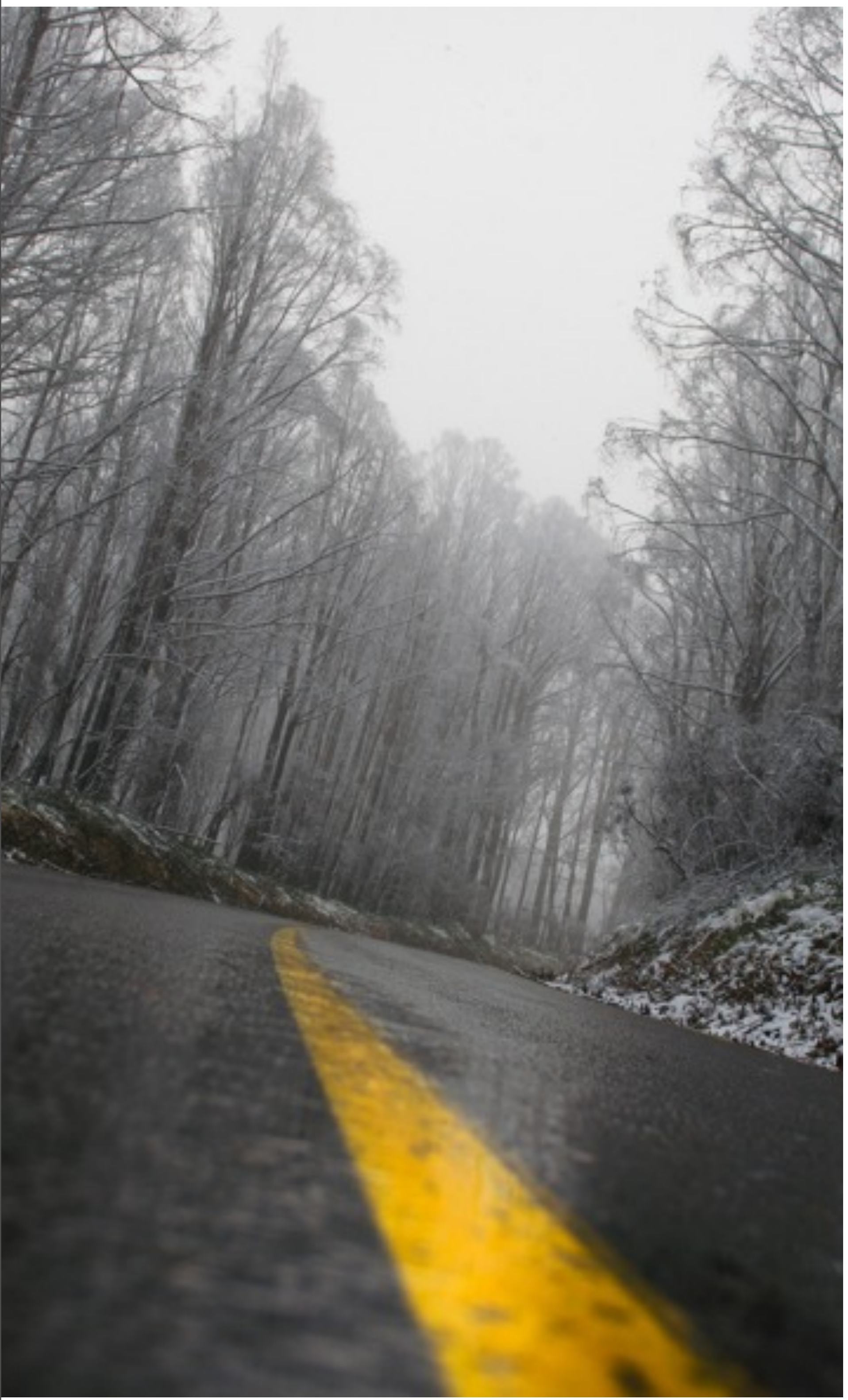
Electrochemical biosensing: The road ahead



• Currently for applications in materials science, there is a growing interest in 'ionogels'.

• Polymers with ionic liquids integrated such that they retain their specific properties within the polymer/gel environment.

Electrochemical biosensing: The road ahead

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Ionogel synthesis:

Inorganic route: Oxides, Sol-Gel. [6]

- Applications in catalysis & photonics.

[6] M.-A. Nouze, J. L. Bideau, P. Gaveau, S. Bellayer and A. Vioux, *Chem. Mater.*, 2006, **18**, 3931-3936.

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Electrochemical biosensing: The road ahead

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Ionogel synthesis:

Inorganic route: Oxides, Sol-Gel.[6]

- Applications in catalysis & photonics.

Organic route: Polymers, Acrylamide gels[7]

- Applications in solid state electrolytes and separations

[6] M.-A. Nouze, J. L. Bideau, P. Gaveau, S. Bellayer and A. Vioux, *Chem. Mater.*, 2006, **18**, 3931-3936.
[7] T. Ueki and M. Watanabe, *Macromolecules*, 2008, **41**, 3739-3749.

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Electrochemical biosensing: The road ahead

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Ionogel synthesis:

Inorganic route: Oxides, Sol-Gel.[6]

- Applications in catalysis & photonics.

Organic route: Polymers, Acrylamide gels[7]

- Applications in solid state electrolytes and separations

[6] M.-A. Nouze, J. L. Bideau, P. Gaveau, S. Bellayer and A. Vioux, *Chem. Mater.*, 2006, **18**, 3931-3936.
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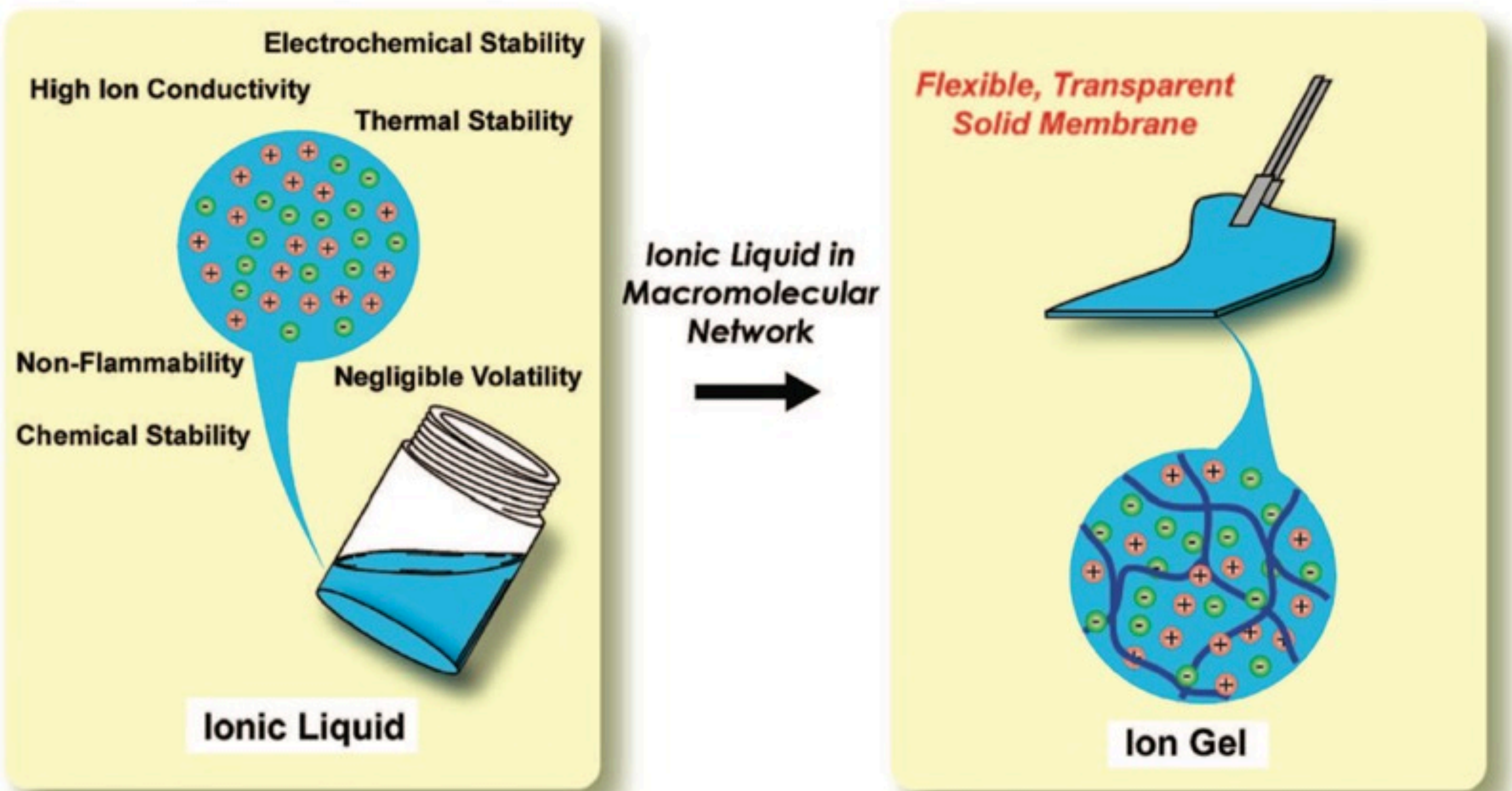
Electrochemical biosensing: The road ahead

Ionogel synthesis:



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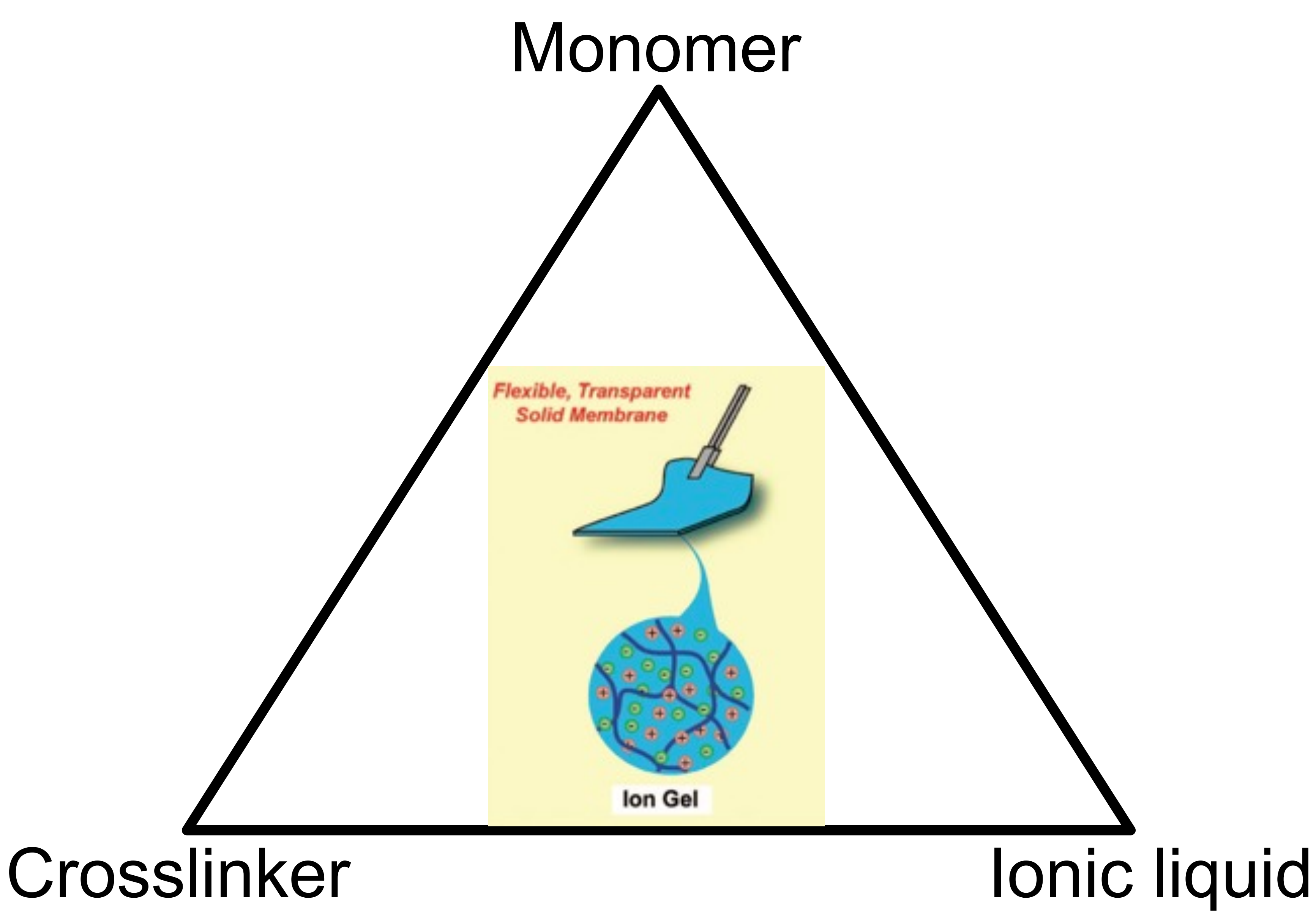
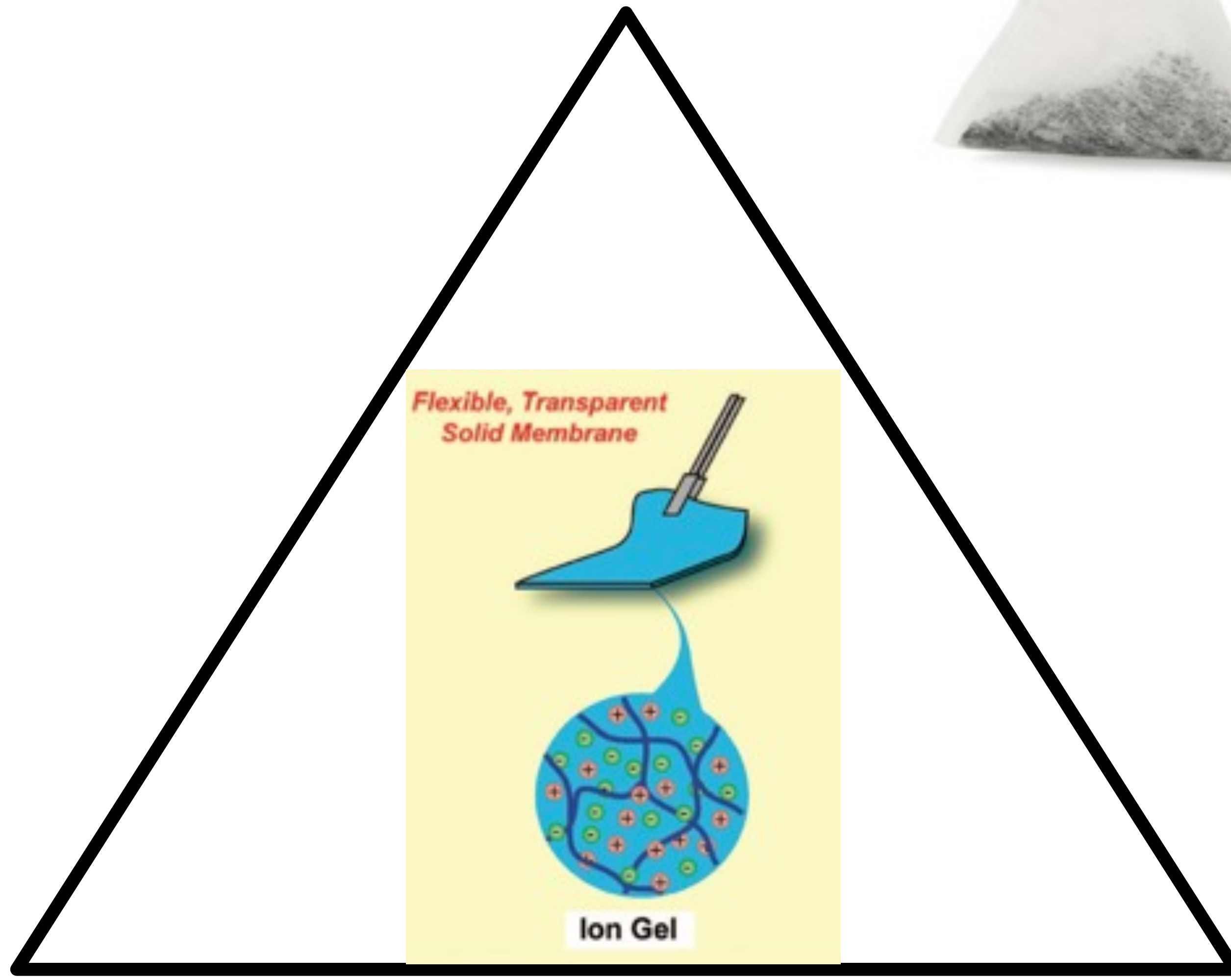
Ionogel synthesis: Organic route



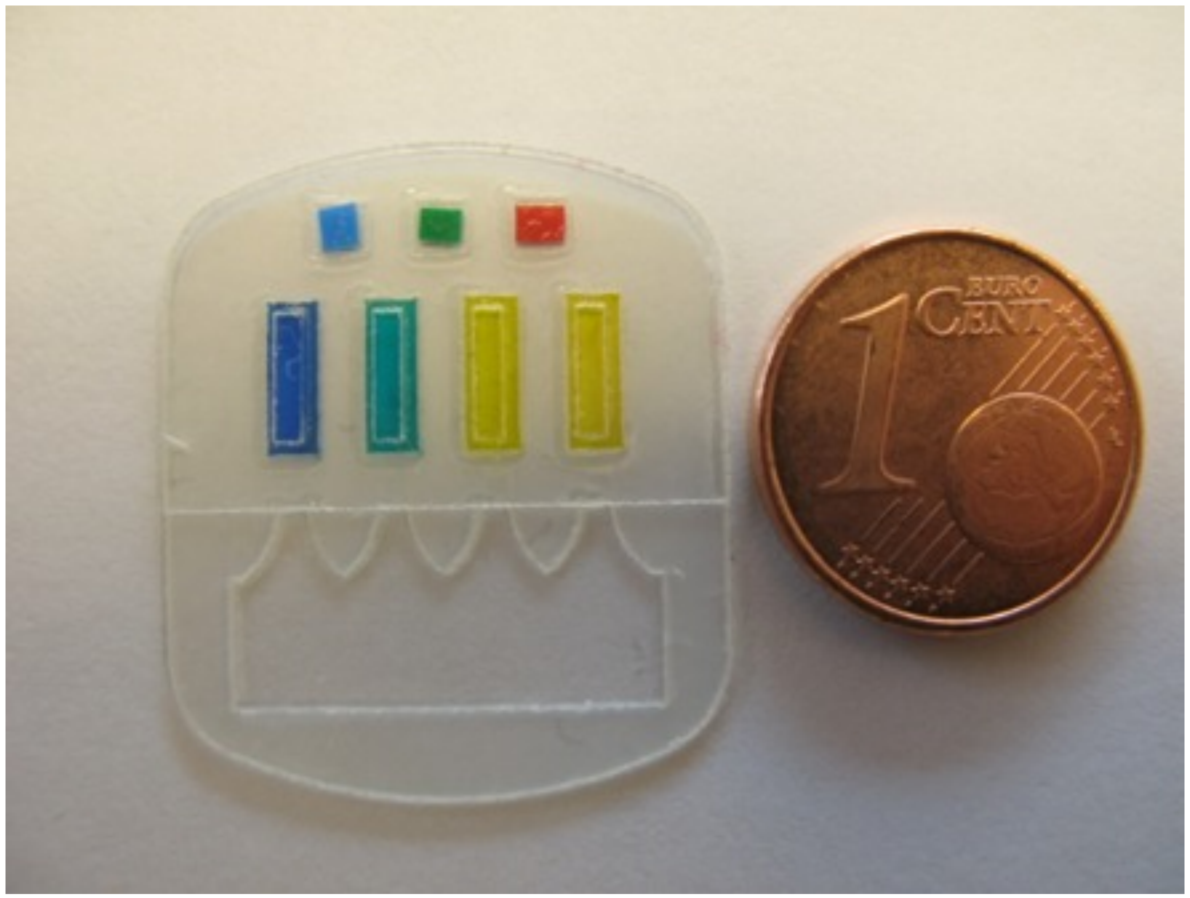
Electrochemical biosensing: The road ahead

Ionogel synthesis: Organic route



Ionogels: Ones we prepared earlier

Sweat Sensing



pH

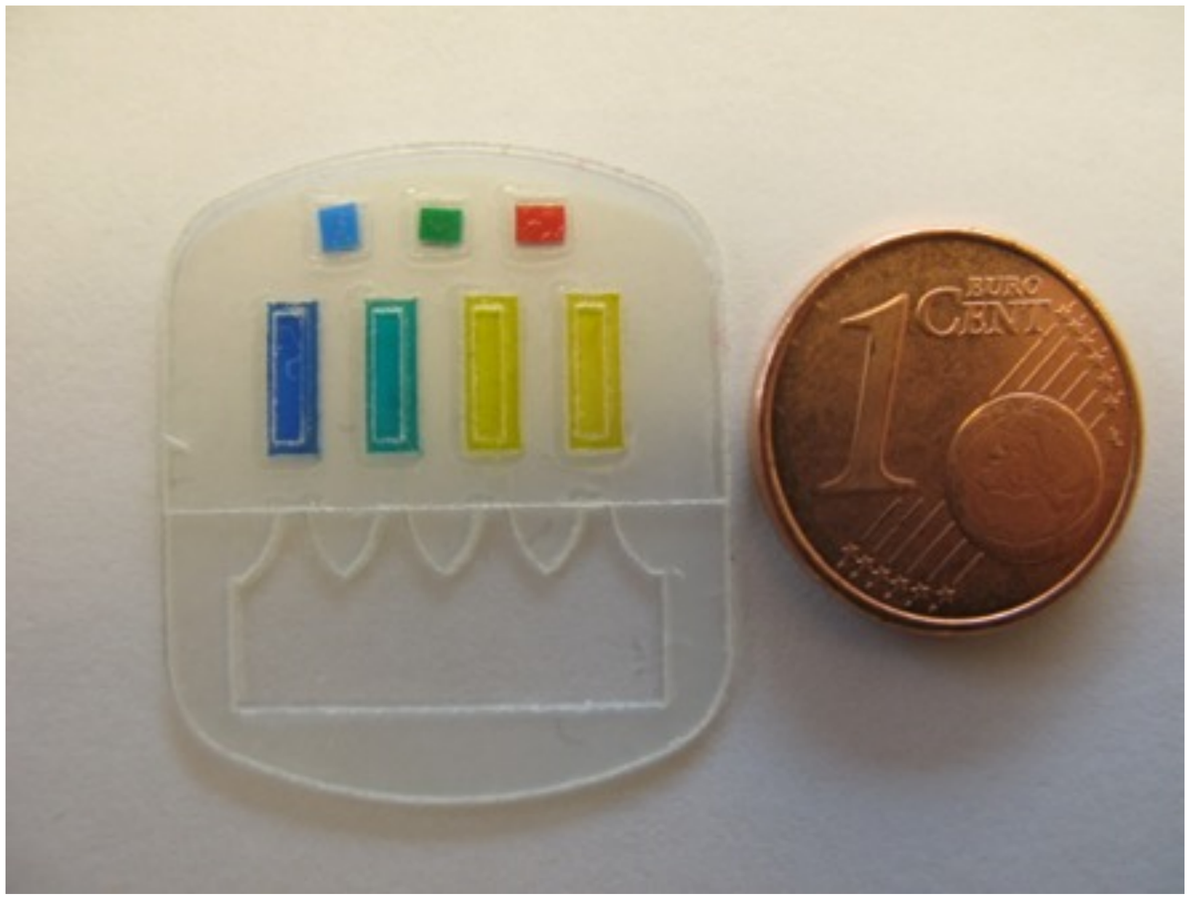
1- BROMOPHENOL BLUE	3.0	4.6
2- BROMOCRESOL GREEN	3.8	5.4
3- BROMOCRESOL PURPLE	5.2	6.8
4- BROMOTHYMOL BLUE	6.0	7.6

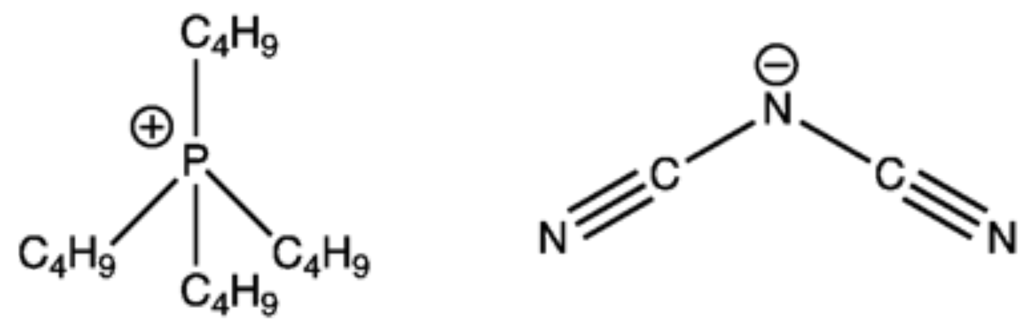
Characteristics:

- Ionogel/dyes
- Image data analysis
- Absorbent = passive pump
- Electronics is not required

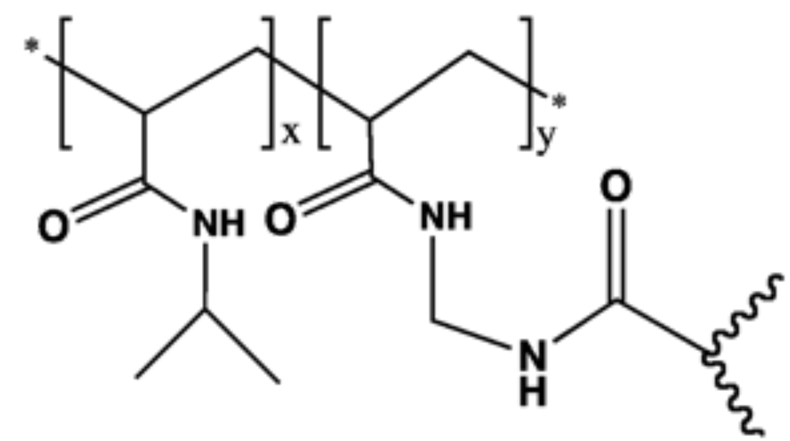
Ionogels: Ones we prepared earlier

Sweat Sensing





$[P_{4,4,4,4}][dca]$



poly(*N*-isopropylacrylamide-co-*N*,*N'*-methylenebisacrylamide)

	pH	
1- BROMOPHENOL BLUE	3.0	4.6
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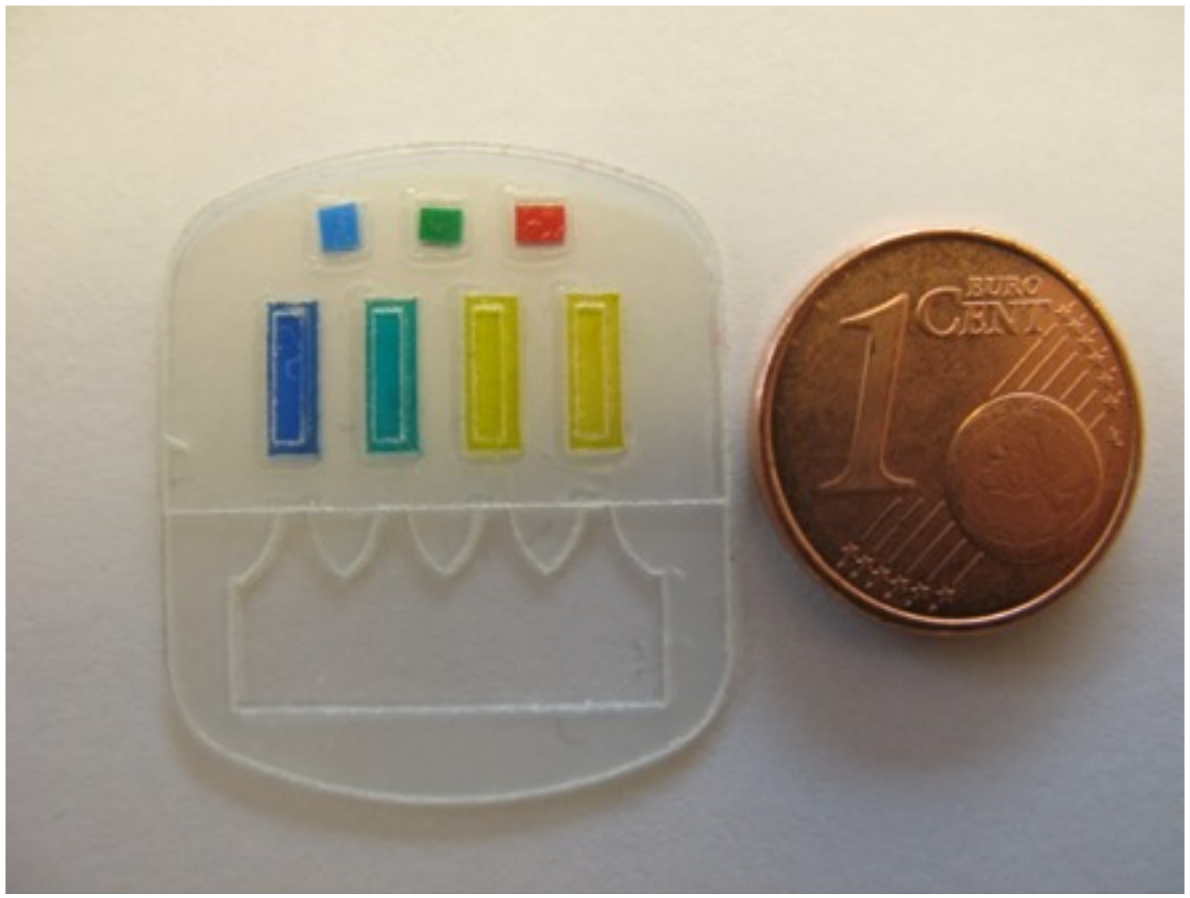

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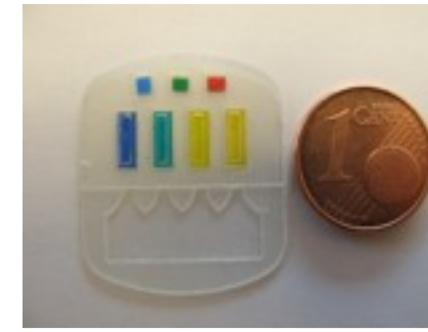
Ionogels: Ones we prepared earlier



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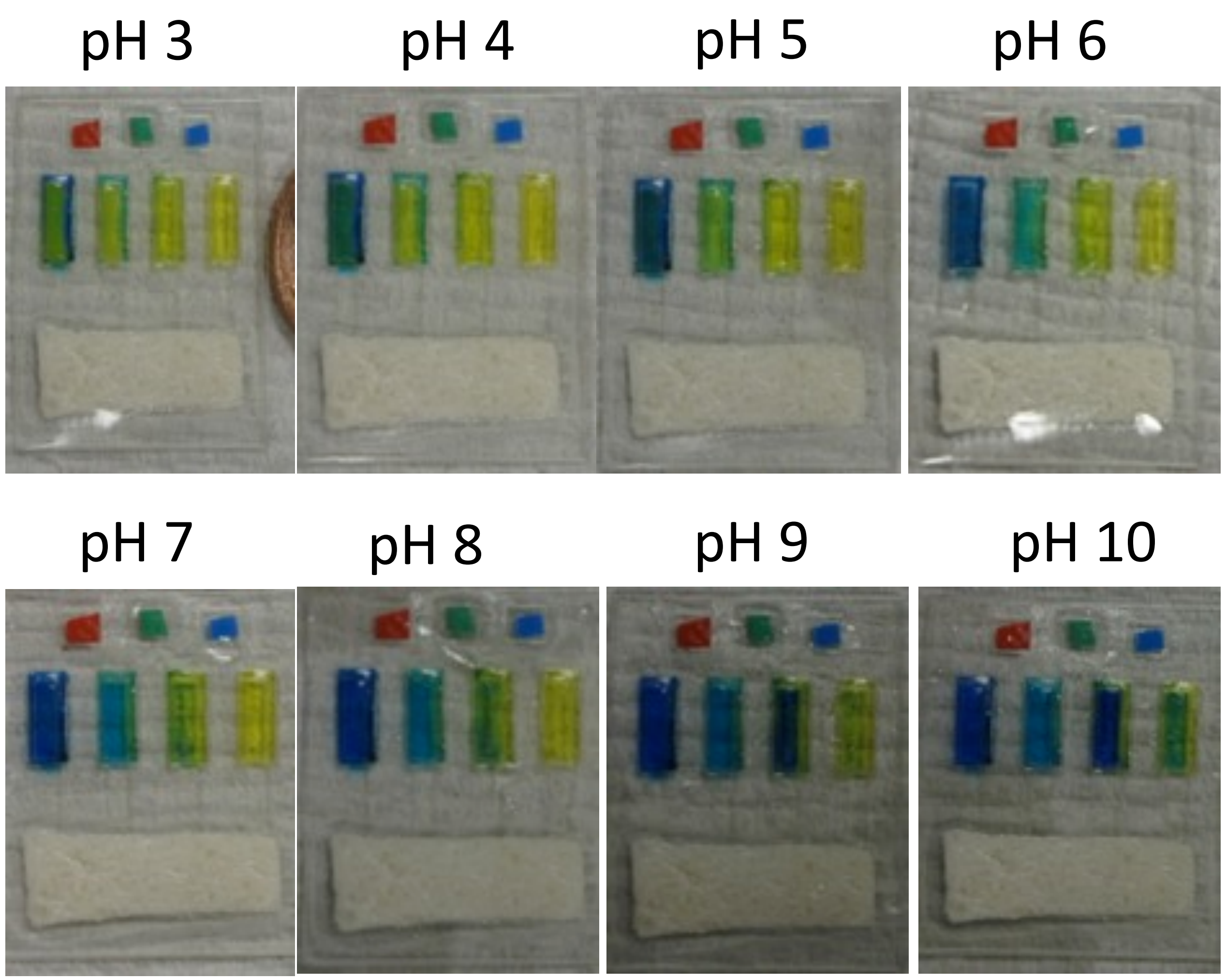

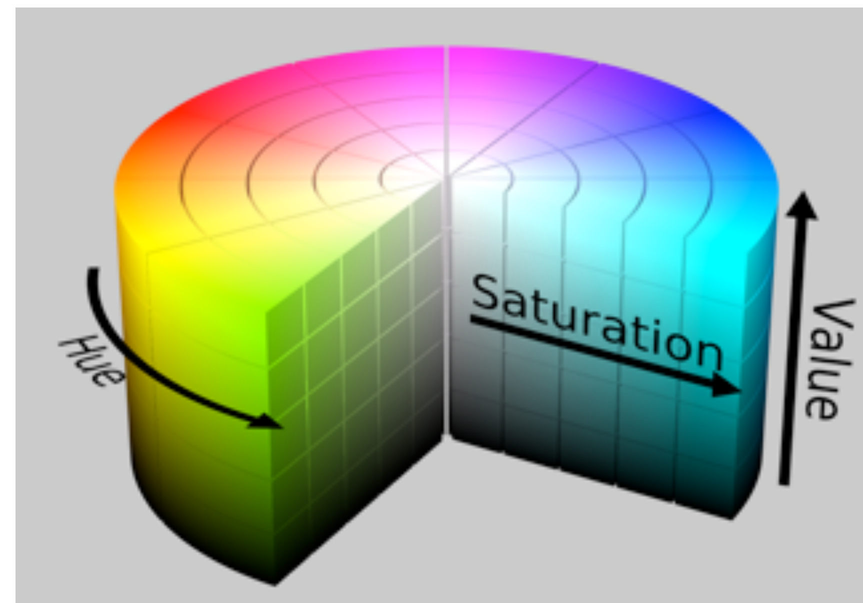

Hue

"the degree to which
a stimulus can be described"

Electrochemical biosensing: Lactate

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• Detection of lactate (deprotonated form of lactic acid) in blood provides a biochemical indicator of anaerobic metabolism in patients with circulatory failure. [8]

• Lactate also found in sweat (concentration range between 9 - 23 mM) [9]

• Lactate concentration increases during physical exercise.

Current detection methods

Electro-chemiluminescent

Carbon nanotubes

Commercially available : SCOUT (sensilab) system

[8] M. H. Weil and A. A. Afifi, Circulation, 41, 989-1001 (1970).

[9] J. M. Green, R. C. Pritchett, T. R. Crews, J. R. McLester and D. C. Tucker, European Journal of Applied Physiology, 91, 1-6 (2004).

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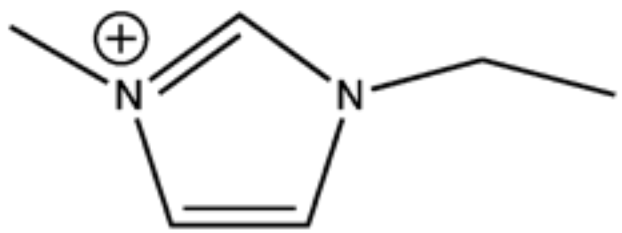
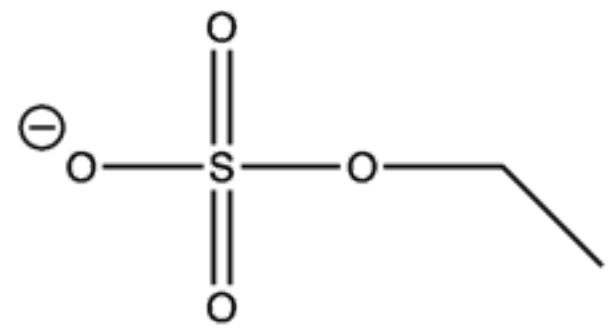
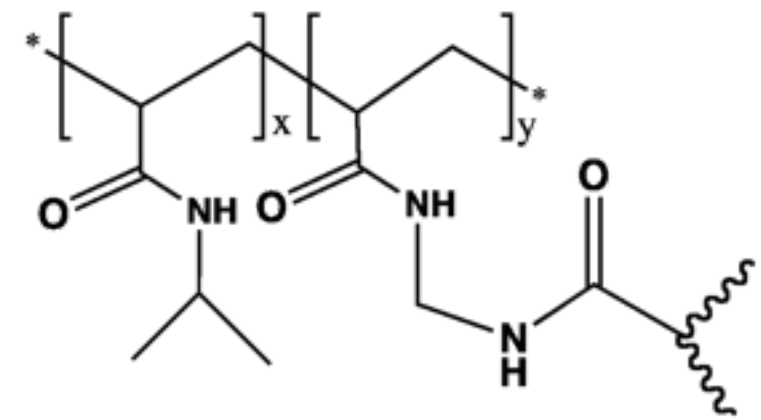
Electrochemical biosensing: Lactate



1-Ethyl-3methylimidazolium $[C_2mIm]^+$



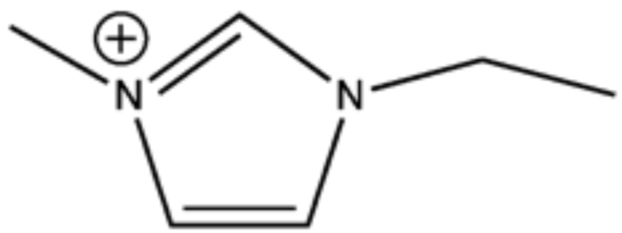
ethyl sulfate $[EtSO_4]^-$



poly(*N*-isopropylacrylamide-co-*N*,*N'*-methylenebisacrylamide)

- Hydrophilic IL chosen due to miscibility with water.
- Avoid mixing problems with PBS & analyte solution.
- Mix the RTIL (17 % water) and PBS solution containing LOx enzyme ratio 4:1
- Hydrated Il completely dissolved the protein with no precipitation observed
- 20 uL final solution drop cast over OECT
- UV polymerised for 1 min

Electrochemical biosensing: Lactate



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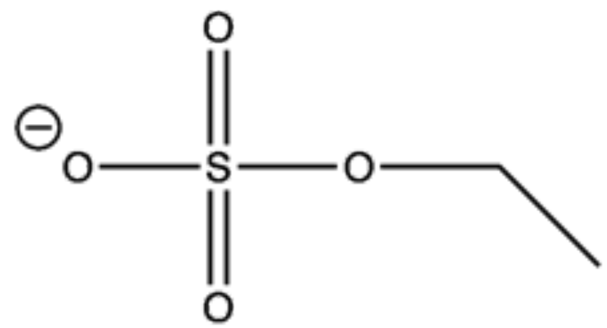

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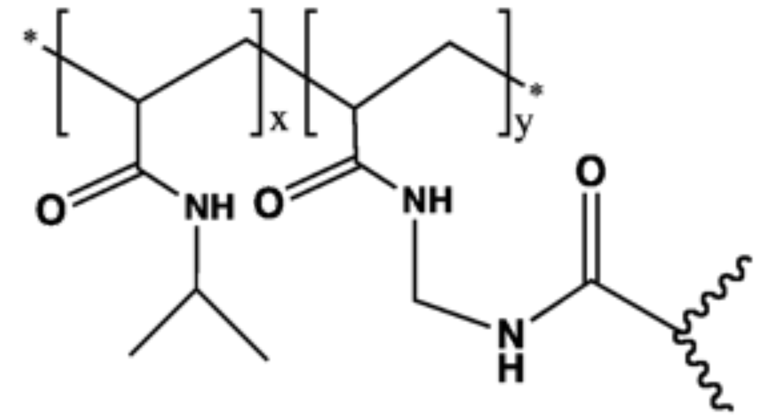
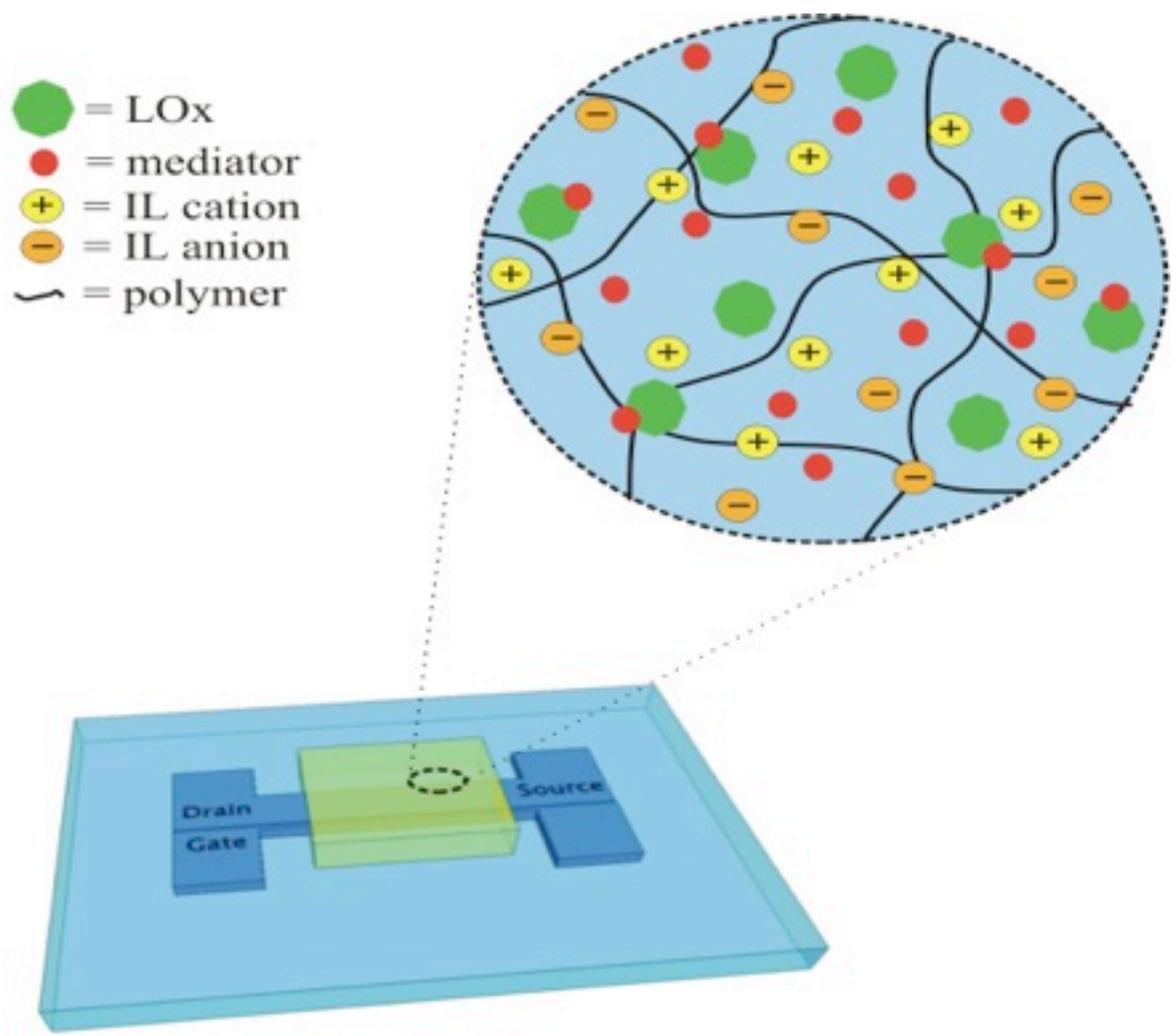


poly(*N*-isopropylacrylamide-co-*N*,*N*'-methylenebisacrylamide)



Electrochemical biosensing: Lactate

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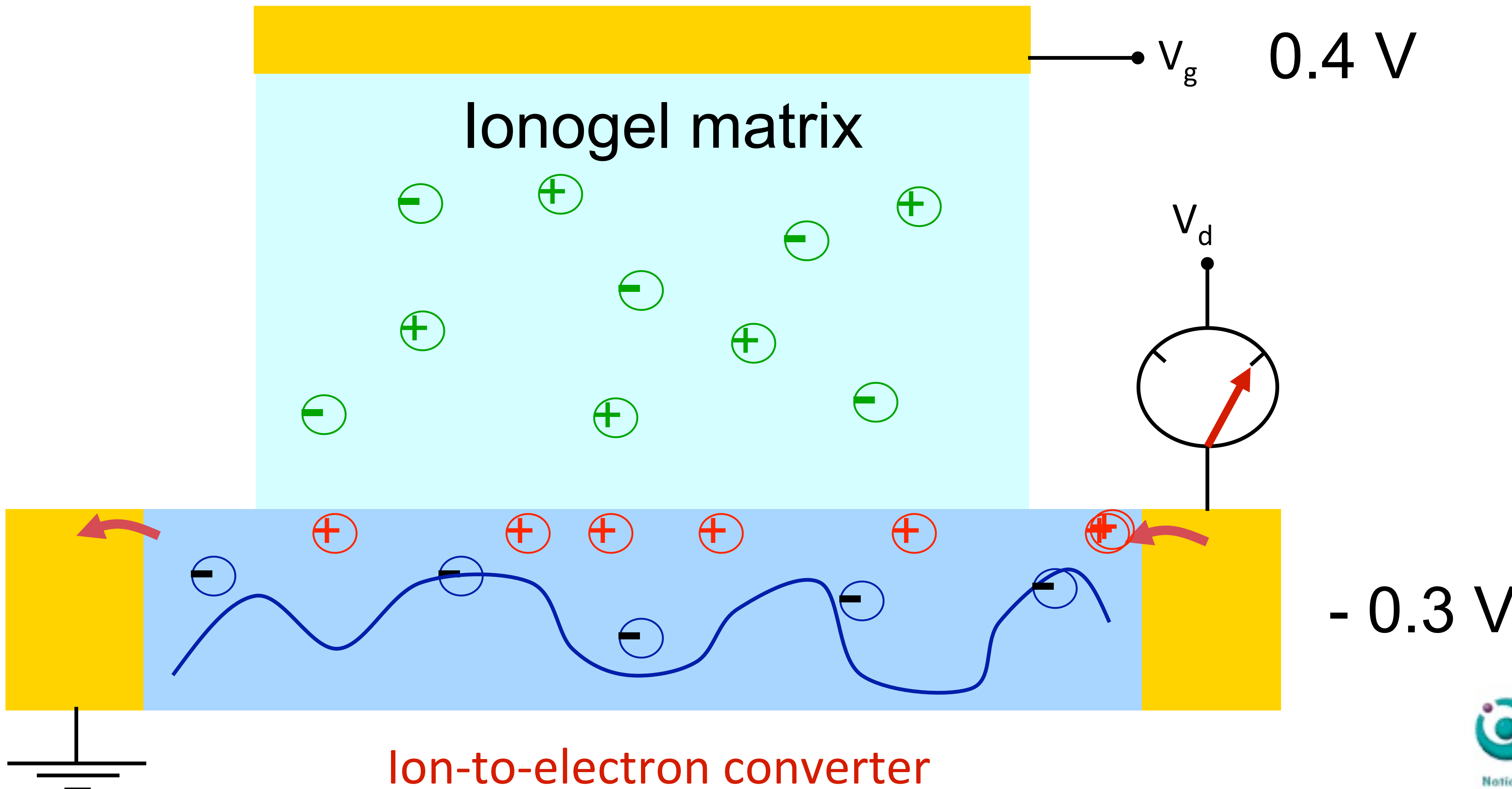
- Apply -0.3 V across the Channel
- Trigger the gate electrode at 0.4 V (3 min square wave pulses)
- Introduce 20 uL of PBS solution with desired lactate concentration



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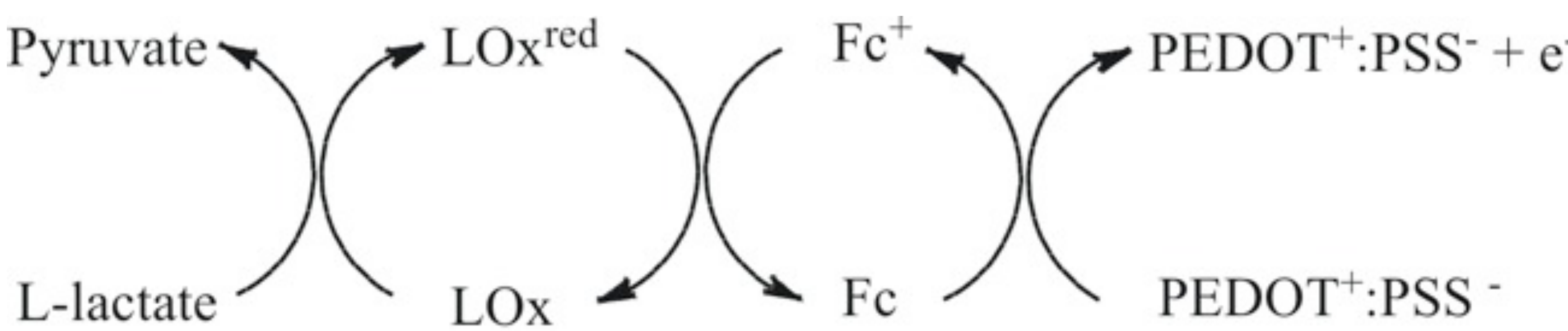
Electrochemical biosensing: Lactate

- Lactic acid is oxidised to pyruvate and cycles back by the Ferriconium ion which carries electrons to the gate electrode

- This leads to a decrease in the potential across the gate / ionogel interface and an increase of the potential at the channel / ionogel interface



} Gate Electrode

$$PEDOT^{+}:PSS^{-} + M^{+} + e^{-} \longrightarrow PEDOT + M^{+}:PSS^{-}$$ } Drain Electrode

As a result more cations from the solution enter the dedoped channel and the modulation of the drain current in response to a voltage increases.

Electrochemical biosensing: Lactate

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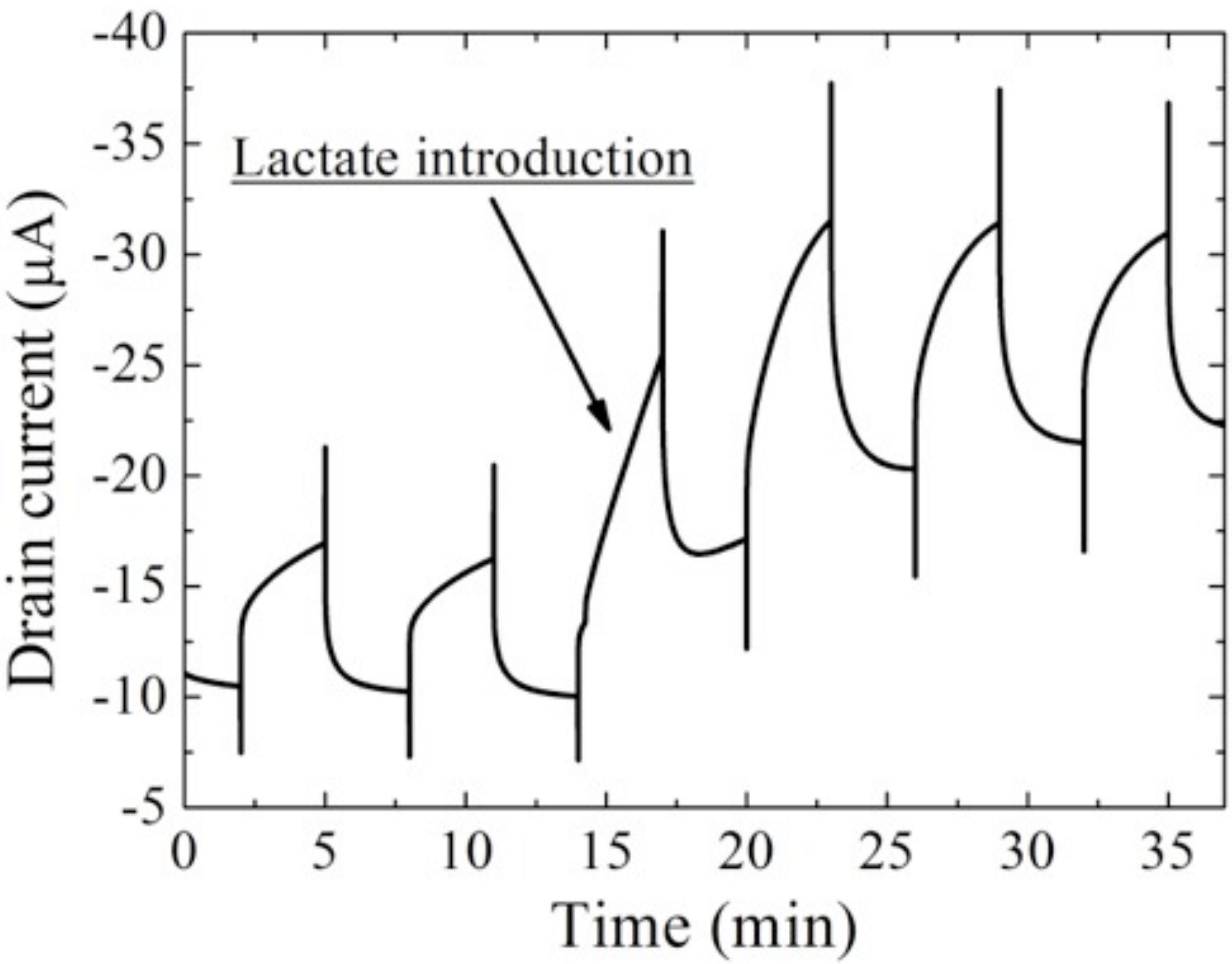
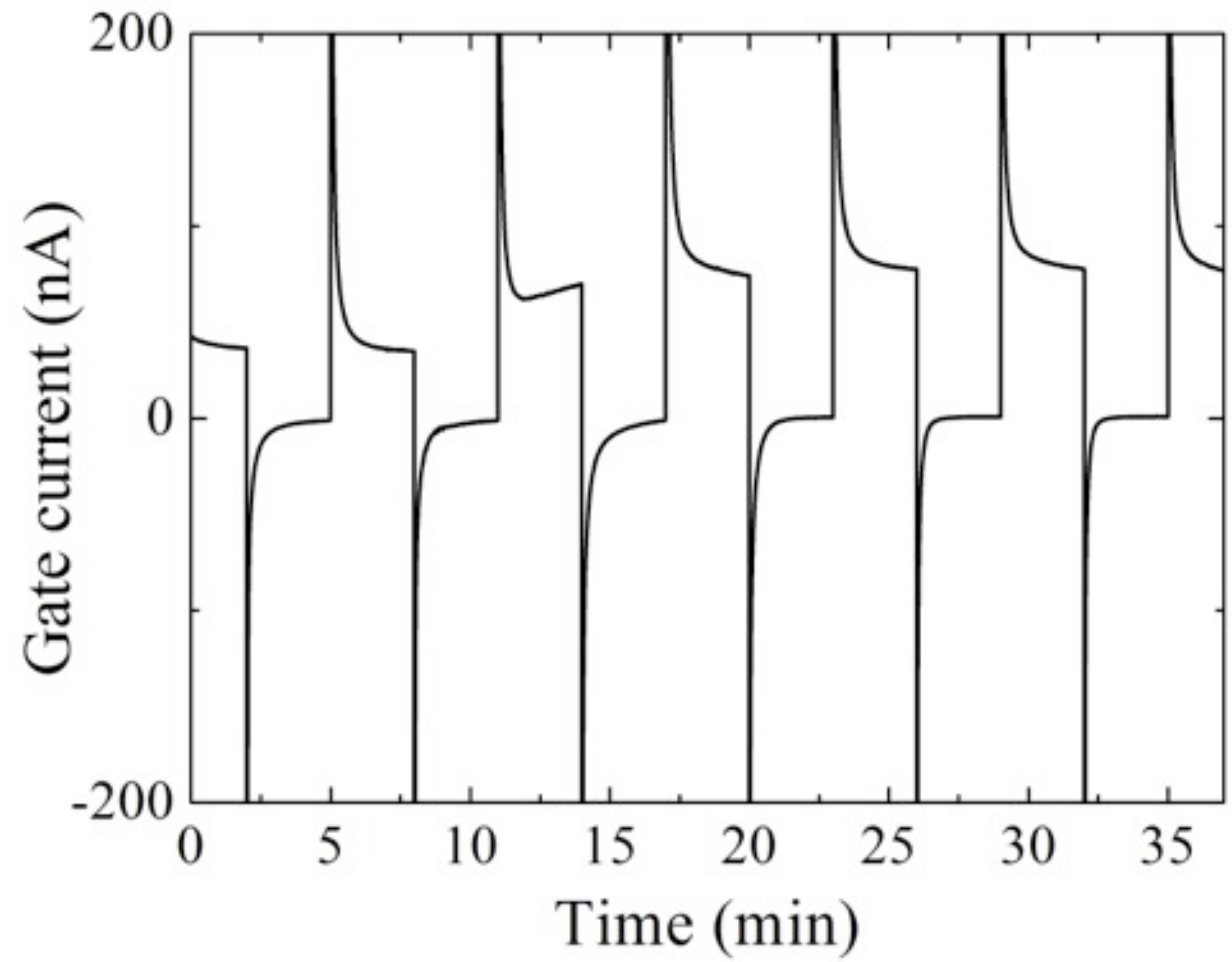
- Modulation in the drain current is much larger than the gate

    100 nA of current at gate - 11 uA at the Drain





- 10 mins for steady state to be reached after analyte added (diffusion inhibited)

[10] D. Khodagholy, V. F. Curto, K. J. Fraser, M. Gurfinkel, R. Byrne, D. Diamond, G. G. Malliaras, F. Benito-Lopez and R. M. Owens, J. Mater. Chem., 22, 4440 (2012).

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Electrochemical biosensing: Lactate

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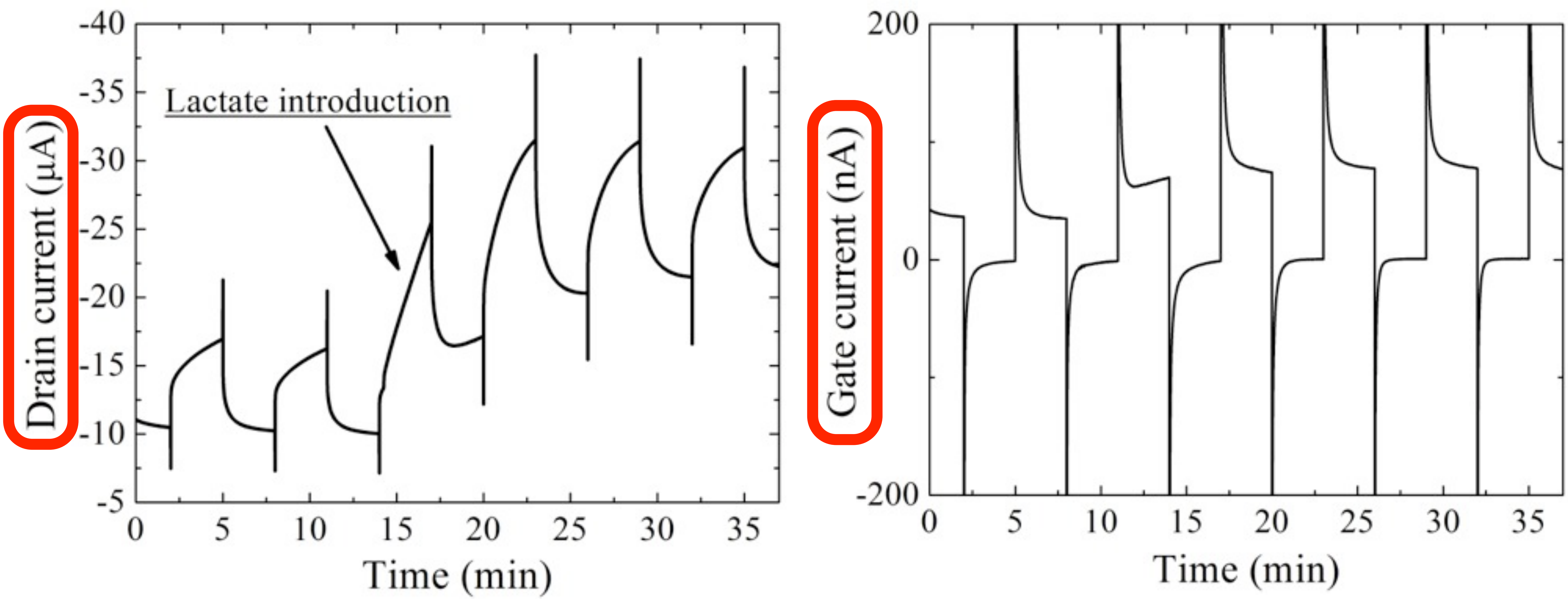

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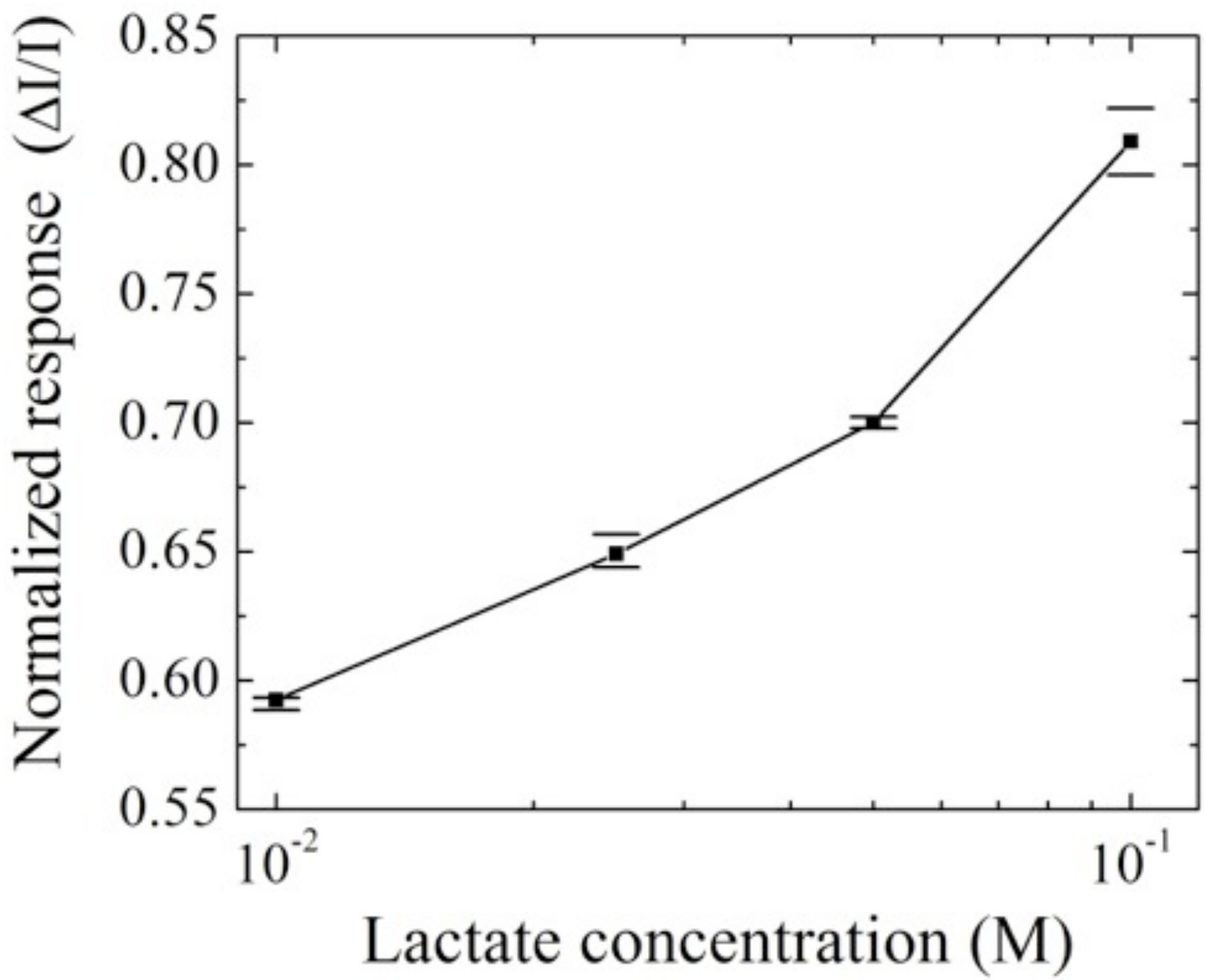

- Response from 10 mM - 100 mM
- In concentration range of sweat

- Compatible with detection of lactate in blood (1.3 - 24 mM)

[10] D. Khodagholy, V. F. Curto, K. J. Fraser, M. Gurfinkel, R. Byrne, D. Diamond, G. G. Malliaras, F. Benito-Lopez and R. M. Owens, J. Mater. Chem., 22, 4440 (2012).

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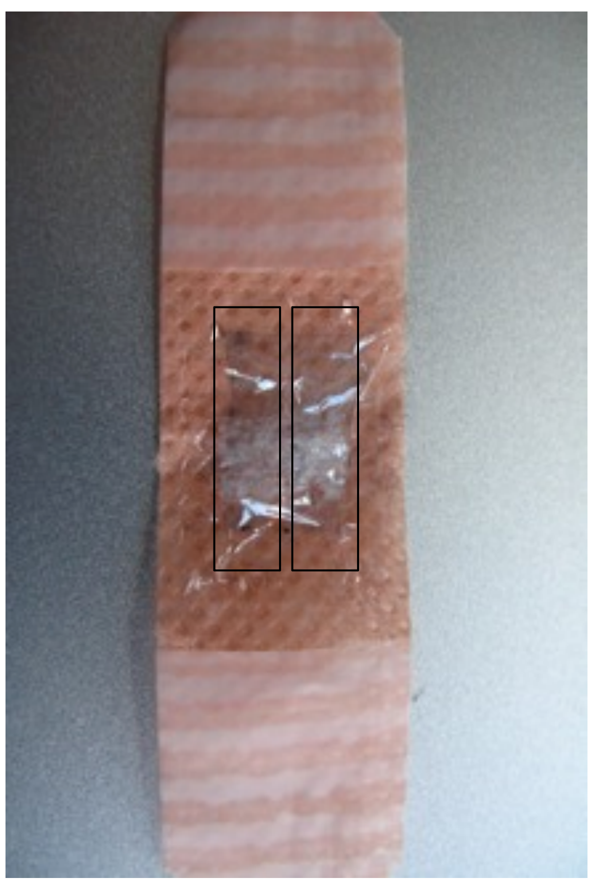
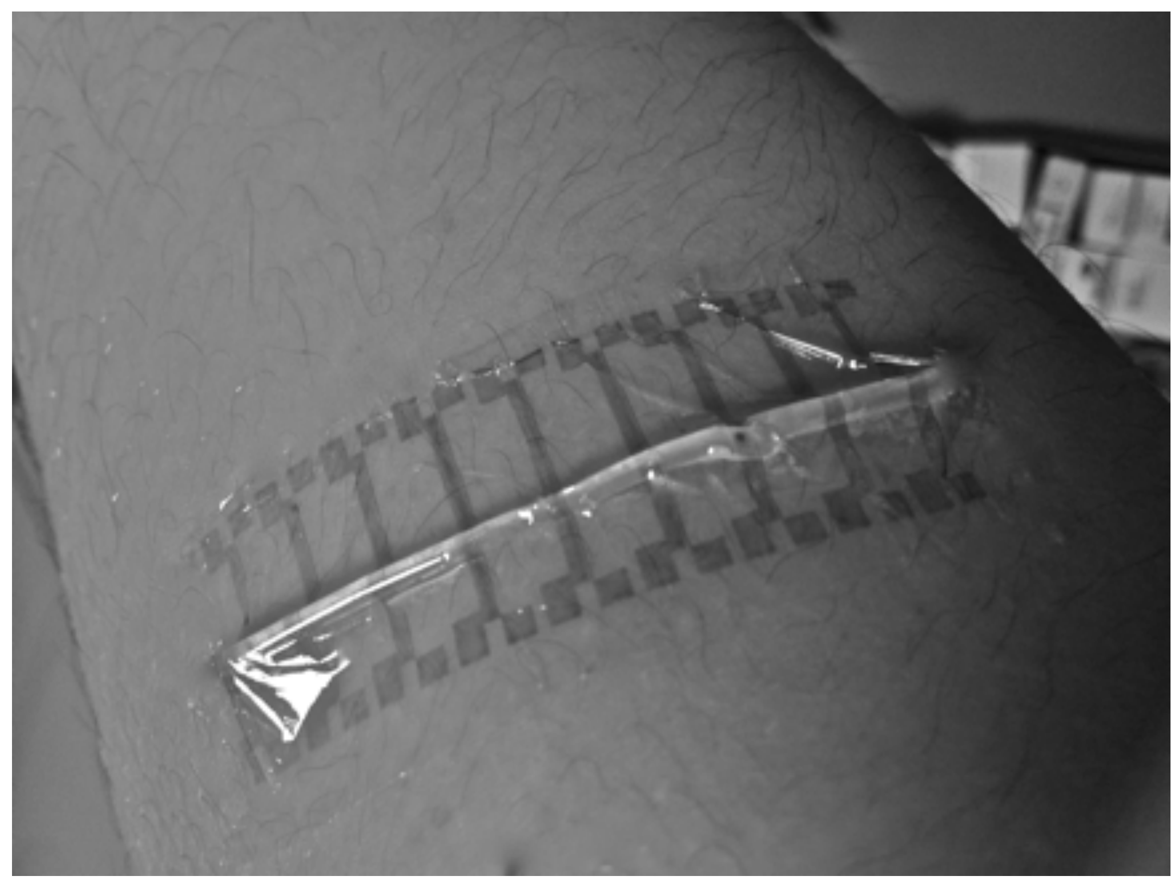
Conclusions

- Detection of lactate in a relevant physiological range (10 - 100 mM)
- Novelty lies in the configuration of the sensor
- Solid State electrolyte on a flexible transistor based biosensor.
  - Implications for the wearability of the sensor

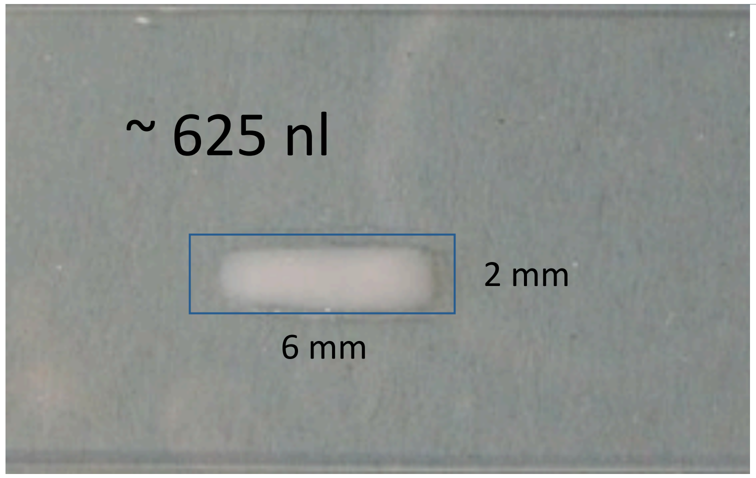




So whats next?

- Enzyme stability and reliability of the system needs to be establish
- Incorporate printable formulations onto OECTs for biosensing

Printing Ionogel



150 um thickness

- Biocompatible Ionogels: pH buffered gels for improved enzymatic shelf life.
- Incorporate OECT / Ionogel into a wireless communicated microfluidic device.

Acknowledgments

Ecole Nationale Supérieure des Mines SAINT-ETIENNE

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Microelectronics Center of Provence
www.emse.fr

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Prof George Malliaras

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Dr Robert Byrne

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Prof Doug MacFarlane

Dr^2 Vijay Ranganathan

Acknowledgments

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Dr Sang Yoo
Prof Róisín C
Prof George I

o F. Curto
ando Benito-Lopez
rt Byrne
rmot Diamond

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SEVENTH FRAMEWORK PROGRAMME

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CLARITY Centre & Ecosystem

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NEUROTESTING

SOCIAL/AGENCY COLLABORATORS

CSET CORE

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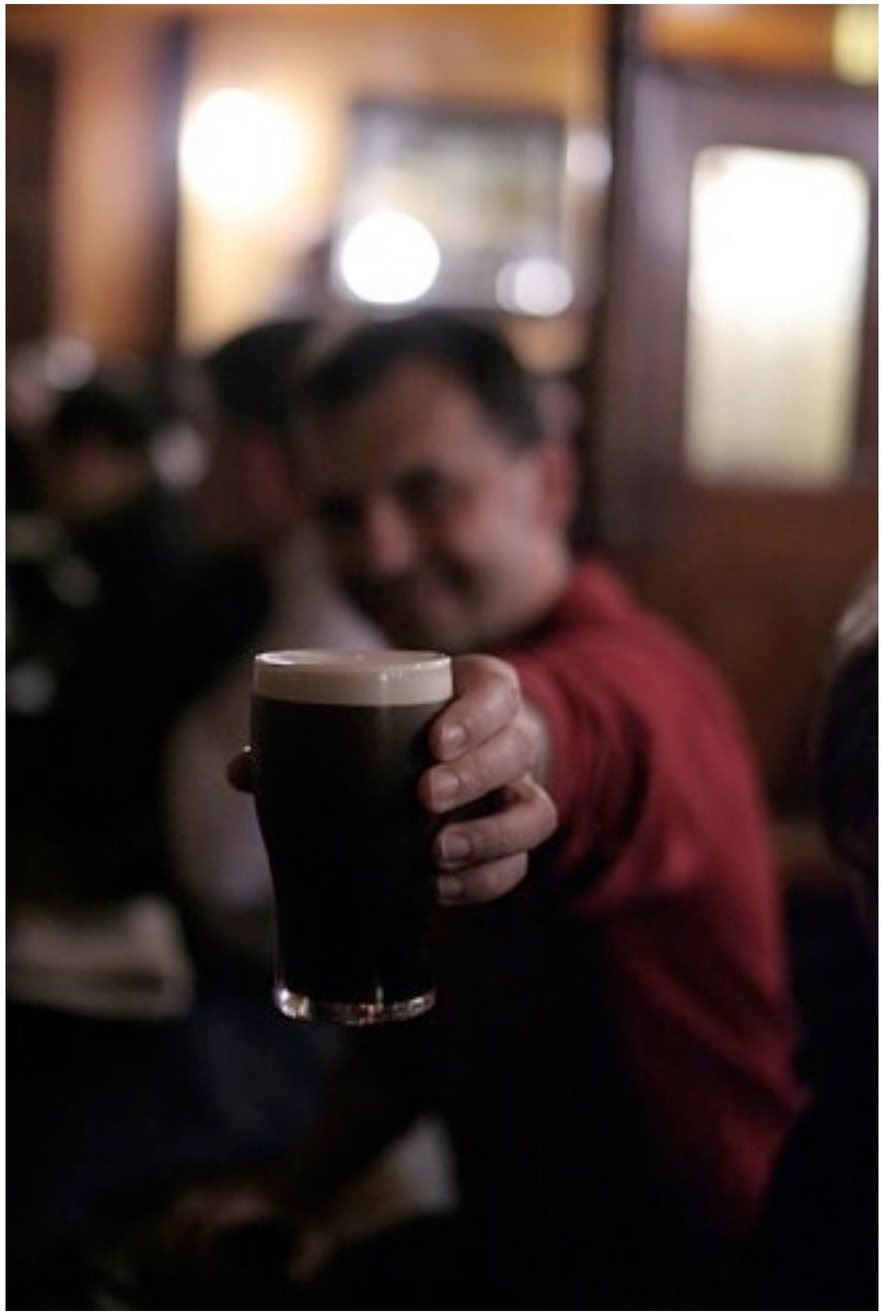

QUESTIONS?

Presentation available for download (@Cyrusmekon)

http://tinyurl.com/7pk4gnb

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